CDM Camp Dresser & McKee

Report

SOUTHEAST ROCKFORD GROUNDWATER CONTAMINATION PHASE II QUALITY ASSURANCE PROJECT PLAN

MARCH 1993

approved
4/7/93

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MARCH 1993

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QUALITY ASSURANCE PROJECT PLAN ADDENDUM
PHASE II: REMEDIAL INVESTIGATION
SOUTHEAST ROCKFORD SITE, ROCKFORD, ILLINOIS
MARCH 1993

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LIST OF ABBREVIATIONS

CDM	Camp Dresser & McKee
CLP	Contract Laboratory Program
CRQL	Contract Required Quantitation Limits
EMSL	Environmental Measurements System Laboratory (USEPA)
FM	Field Manager
FS	Feasibility Study
GC/MS	Gas Chromatography/Mass Spectroscopy
HSP	Health and Safety Plan
IDPH	Illinois Department of Public Health
MCL	Maximum Contaminant Level
OVA	Organic Vapor Analyzer
PRS	Proposed Illinois Groundwater Quality Standards
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RAL	Remedial Action Level
RAS	Routine Analytical Services (CLP)
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
RPM	Remedial Project Manager (USEPA)
RPO	Regional Project Officer (USEPA)
RSCC	Regional Sample Control Coordinator
SAP	Sampling and Analysis Plan
SAS	Special Analytical Services (CLP)
SDWA	Safe Drinking Water Act
SIPM	Site Investigation Procedures Manual

LIST OF ABBREVIATIONS

(Continued)

SM	Site Manager
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyte List
TAT	Technical Assistance Team
TCL	Target Compound List
USEPA	U.S. Environmental Protection Agency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WA	Work Assignment

1.0 PROJECT DESCRIPTION

The Southeast Rockford Groundwater Contamination Phase II investigation will involve a source investigation, groundwater investigation, residential well sampling and residential indoor air sampling for the purpose of locating contaminant source areas, defining the horizontal and vertical extent of the groundwater plume and to evaluate feasible remedial alternatives and/or the need for additional studies.

1.1 STUDY AREA BACKGROUND

The study area for Phase II is located in the southeast part of Rockford in Winnebago County and covers approximately 10 square miles. The study area is bounded by Broadway to the north, Wendy Lane to the east, Sandy Hollow Road to the south, and the Rock River to the west. The study area is shown in Figure 1-1.

The study area has been expanded northward from Harrison Avenue to the Broadway boundary because sampling results have indicated that source areas may exist outside the original site boundaries. It also appears that the contamination plume extends beyond the original Phase I study area.

The history of the Southeast Rockford Superfund site is included in CDM's Phase I and Phase II Work Plans. From June to October of 1991 CDM and its subcontractors performed Phase I field investigations under the direction of the Illinois Environmental Protection Agency (IEPA). These investigations included drilling and installing groundwater monitoring wells, well surveying, hydraulic conductivity testing, geophysical logging, soil gas surveying, and soil and groundwater sampling.

STUDY AREA BOUNDARY

N

BROADWAY

BROADWAY

KISHWAUKEE STREET

ELEVENTH ST

TWENTIETH ST

ALPINE ROAD

CHARLES ST

MULFORD ROAD

HARRISON AVENUE

HARRISON AVENUE

HARRISON AVENUE

CMSP&P RR

KISHWAUKEE STREET

ELEVENTH STREET

CMSP&P RR

BN RR

TWENTIETH STREET

GREEN DALE DRIVE

SILENT WOOD TRAIL

26TH AVENUE

EASTROCK DRIVE

PENNY LANE

BALSAM LANE

BAVARIAN LANE

O'CONNELL ST

ALPINE ROAD

AMERICAN ROAD

PYRAMID DRIVE

MAYFLOWER

ROCK RIVER

SANDY HOLLOW ROAD

SANDY HOLLOW ROAD

SANDY HOLLOW ROAD

SCALE:

500 0 1000 Feet

SOUTHEAST ROCKFORD GROUNDWATER CONTAMINATION STUDY

STUDY AREA

CDM

environmental engineers, scientists, planners, & management consultants

US4247

Figure No. 1-1

D:\1681\7\RI\PLN\ FIG1-1 03/08/93 G. FOSZCZ

Data from the Camp Dresser & McKee (CDM) Phase I Investigation (June to October 1991) indicates two major areas of groundwater contamination of volatile organic compounds (VOCs) located within the Phase I study area.

According to the Phase I results, significant levels of 1,1,1-trichloroethane (TCA), 1,1-dichloroethane (1,1-DCA), and 1,1-dichloroethene (1,1-DCE), were detected in a relatively small area centered near the industrial facility southeast of the intersection of Harrison Avenue and Alpine Road, and significant levels of trichloroethene (TCE), TCA, cis-1,2-dichloroethene (cis-1,2-DCE), and 1,1-DCA were detected in a large area near and downgradient (west-northwest) from well nest MW106. Also, several smaller plumes located west and southwest of MW20 indicated the presence of widespread chlorinated organic compounds, including TCA, TCE, PCE, cis-1,2-DCE, 1,1-DCA, and 1,1-DCE, in the groundwater. A summary of existing data is presented in Appendix A.

Though several localized detections were reported during the Phase I investigations, there is no evidence in the groundwater of significant migration of semi-volatiles, pesticides, or most inorganic contaminants. Also, recorded exceedances of secondary MCLs by aluminum, iron, and manganese in groundwater within the study area probably reflect a combination of natural concentrations and localized contamination. As a result, for purposes of plume characterization during the Phase II investigation (accomplished in the groundwater investigation portion of this project, as detailed in section 1.2.2), groundwater analysis will be limited to volatile organic compounds. For the purposes of source identification and characterization, groundwater samples will be analyzed for Target Compound List (TCL) Organics, Target Analyte List (TAL) Inorganics, minerals, and nutrients (accomplished in the source investigation portion of the project, as detailed in section 1.2.1).

The Phase I groundwater sampling results suggest potential source areas corresponding to significant contamination in the following areas: 1) upgradient from well nest MW106 (Area 7); 2) upgradient from well nest MW101 (Area 5); 3) at the industrial facility southeast of

Harrison Avenue and Alpine Road (Area 8); and 4) several discrete locations in the western part of the study area (Areas 1 through 4).

1.2 PROJECT SCOPE AND OBJECTIVES

As stated in the Phase I RI Work Plan, more extensive and comprehensive investigation of the above-mentioned contamination problems will be addressed through a Phase II RI. Principal objectives of the Phase II RI are to:

- Gather historical information on the study area utilizing aerial photographs and IEPA and USEPA files to research industrial or other operations to determine the origin and timing of any contaminant releases to soil or groundwater.

- Conduct a soil gas survey, advance soil borings, excavate test pits, and install monitoring wells to evaluate potential source areas defined in Phase I, as well as additional potential source areas defined since Phase I from information on industrial activities and land use. (The soil gas survey was conducted in winter 1993, and was governed by the Phase I QAPP.)

- Define the vertical and horizontal extent of groundwater contamination throughout that portion of the study area between Sandy Hollow Road and Harrison Avenue; in the recently-added part of the study area between Harrison and Broadway, define contaminant migration pathways between potential source areas and the site.

- Evaluate the potential for any dense non-aqueous phase liquids (DNAPLs) in the subsurface through the collection of soil samples in potential source areas.

- Monitor VOC vapors in residential basements located in areas of elevated contaminant concentrations in shallow groundwater.

- Perform a residential well survey and well sampling program for homes within the operable unit study area that have not been hooked up to public water supply to provide data for the risk assessment.

- Perform groundwater modelling to further define the plume area, predict future contaminant distributions, assist in locating source areas, and assist in evaluating the feasibility of remedial alternatives.

In order to achieve these objectives, CDM will work to obtain the following sets of data:

(1) Detailed site map,

(2) Aquifer characteristics,

(3) Information on horizontal and vertical distribution of groundwater contamination,

(4) Piezometric surface and groundwater flow characteristics in each aquifer,

(5) Contaminant source areas located in the soil, and

(6) Indoor air characterization.

Field work will be conducted as described in Sections 3.5 and 3.6 of the Work Plan and in the following subsections, and will include a source investigation, a groundwater investigation, residential well sampling and residential air sampling. The Phase II source investigation will consist of collection of soil samples from soil borings; location, drilling, and installation of monitoring wells; and collection of groundwater samples. An additional component of the Phase II source investigation includes the soil gas survey that was conducted in winter 1993 under the Phase I QAPP (refer to Work Plan and Sampling and

Analysis Plan for Soil Gas Survey, October 1992). The Phase II groundwater investigation will include the location, drilling, and installation of monitoring wells, and collection of groundwater samples. Residential well sampling will involve collection of groundwater samples from residential wells currently in use. Residential air sampling will include collection of indoor and outdoor ambient air samples. The procedures used to perform these tasks are described in detail in the Sampling and Analysis Plan (SAP).

1.2.1 SOURCE INVESTIGATION

1.2.1.1 Soil Borings and Test Pits

The objective of advancing soil borings is to collect subsurface soil samples to define soil contamination in the vadose zone near potential source areas. Soil borings will be placed in "hot spots" located during the soil gas survey performed in winter 1993. It is anticipated that approximately four borings will be placed in each source area. Two test pits will be excavated in Area 7, which has shown an extensive area of elevated concentrations of VOCs in soil gas. The purpose of the test pits is to determine the character and continuity of subsurface wastes at two locations exhibiting soil gas and/or geophysical anomalies in Area 7.

Soil boring samples will be collected at five-foot intervals from the surface to the water table, and will be analyzed (to the extent possible, depending on sample recovery) for Target Compound List (TCL) organics and Target Analyte List (TAL) inorganics. The samples will be field screened using an organic vapor monitor (OVM). Two samples from each boring will be secured for analysis: the sample with the highest VOC concentrations as measured by head-space, and the first sample below the contaminated zone that shows undetectable VOCs by head-space. The rationale for the selection of these samples is that they will provide general upper and lower bounds for VOC concentrations in each boring. For borings that do not indicate VOC readings from field screening, the soil sample nearest the water table will be secured for analysis.

At test pits, each backhoe bucket will be scanned for VOCs using an HNu, OVM or equivalent, and the contents will be characterized visually. Approximately four samples will be collected from each test pit. Sample selection will be based primarily upon visual characteristics (i.e., soils saturated with product will be selected for analysis), and secondarily upon VOC concentrations (the samples with the highest VOC screening concentrations will be selected). If four samples cannot be selected from a test pit by visual or VOC screening methods, samples will be selected based on geographic coverage, with samples being collected from different portions of the test pit. The analyses to be performed on these samples are TCL organics and TAL inorganics. The two most contaminated samples (based on screening characteristics) will also be submitted for analysis of TCLP organic and inorganic parameters.

Air sampling will be conducted at each test pit, using SKC portable sampling pumps (or equivalent) with Tenax tubes. The SKC sampling pumps will be mounted on tripods and positioned along each side of each pit, and at various distances downwind from each pit. Sampling pumps will be calibrated prior to sampling. Approximately 10 air samples will be collected at each pit (approximately seven downwind and three upwind). Downwind samples will be collected at various distances from the test pits. Duplicate samples will be collected at a frequency of one per 10 samples. The specific sampling procedure for the air samples collected from near the test pits is detailed in "Atmospheric Sampling for Volatile Organic Compounds" in Appendix B of the SAP. The samples will be analyzed by EPA method TO1, which is specified in the SAS Request Forms found in Appendix B of this document.

1.2.1.2 Monitoring Wells: Soil and Groundwater Sampling

The primary objective of installing monitoring wells in the source investigation is to confirm potential source areas. Secondary objectives are to more accurately define the lateral and vertical extent of groundwater contamination, gather additional information for groundwater modeling, further define the local geology and hydrogeology of the study area, and provide data for the risk assessment and for evaluating potential remedial alternatives.

A total of 31 monitoring wells at 23 locations will be installed during the source investigation. Soil samples will be collected during the installation of the wells for analytical, lithologic, and geotechnical purposes. Section 3.6.1.3 of the Work Plan describes the purpose of and methods used for lithologic and geotechnical sampling. Analytical soil samples will be collected at five-foot intervals. However, subsurface soil samples will be submitted for laboratory analysis from only approximately 20% of these wells; of the monitoring wells from which soil samples will be submitted, approximately three samples will be submitted from each well, and samples will be from the saturated zone only. The specific wells to be sampled for analytical purposes will be selected in order to provide data representative of the study area. At each monitoring well sampling location, the samples to be submitted for analysis will be one sample from the screened interval, if possible, and approximately two samples from unconsolidated stratigraphic units different from the screened interval sample. Analyses to be performed on these samples are TCL organics, TAL inorganics, and total organic carbon. The total organic carbon analysis will allow estimation of the partitioning preference of contaminants between the groundwater and the solid materials of the aquifer.

Groundwater Samples Collected During Drilling

In order to determine the optimal depth interval at which to set the monitoring well screen, groundwater samples will be collected at 10-foot intervals during drilling at selected monitoring well locations (in both source and groundwater investigations). These samples will be submitted to a local laboratory subcontracted to CDM for 24-hour analysis of VOCs. Upon receipt of the analytical results, the field geologist will determine the depth interval at which the monitoring well screen will be set. The depth interval will be centered on the depth of the sample with the highest VOC concentrations reported for the particular drilling location.

Collection and analysis of groundwater samples during drilling will allow elimination of one or more monitoring wells at certain well nest locations, and allow monitoring to focus on the

specific depth of peak contaminant concentrations. The collection of groundwater samples during drilling will generally be limited to locations which are: 1) more than 500 feet from potential contaminant source areas; and 2) in areas where the pattern of contamination with depth is unknown. The source investigation monitoring well locations to be sampled for groundwater during drilling are wells MW119 through MW124.

Groundwater Sampling from Monitoring Wells

Samples of groundwater from the 31 monitoring wells installed during the source investigation will be collected and analyzed for target compounds list (TCL) organics and target analyte list (TAL) inorganics. Because groundwater is currently used as a potable water source in the area, lower detection limits will be required for VOCs to allow a comparison with drinking water standards. Selected wells (20% of all wells to be sampled; the selected wells will be representative of the contaminant plumes in the various aquifers) will be analyzed for general water chemistry parameters including minerals (alkalinity, fluoride, chloride, sulfate, and silica) and nutrients (COD, ammonia, total kjeldahl nitrogen, nitrate, nitrite, total phosphorus, and TOC), as well as total dissolved solids and total suspended solids for remedial design purposes. These parameters are useful in evaluating most conventional remedial treatment methods for contaminated groundwater, including air stripping, carbon adsorption, bioremediation, and chemical treatment methods. The wells to be sampled for these analyses are listed in Table 5-5 of the Sampling and Analysis Plan (SAP).

1.2.2 GROUNDWATER INVESTIGATION

The primary objectives of the Phase II groundwater investigation are to better define the lateral and vertical extent of the groundwater contaminant plume, to better define the local geology and hydrogeology of the study area, and to provide information that can be used in groundwater modeling, in risk assessment, and for evaluating potential remedial alternatives.

These objectives apply primarily to that portion of the study area south of Harrison Avenue. North of Harrison, few residents are or were supplied by private wells. As a result, the primary objective of the Phase II study in this area is to define whether and how contaminants migrate toward the site from identified sources within this area.

The 19 new wells installed at nine locations for the Phase II groundwater investigation will be screened at different depths at a given location to provide vertical profiles of contaminant concentrations at the location, from near the water table to depths as great as 300 feet below the water table (325 feet below ground surface). In the western portion of the study area, the maximum depth of contamination is not known, while in the eastern part it extends at least 70 feet into the bedrock aquifer (and 140 feet below ground surface).

1.2.2.1 Soil Samples

Subsurface soil samples will be taken during the installation of the monitoring wells for the groundwater investigation. These samples will be taken as described in Section 3.6.1.3 of the Work Plan. Section 1.2.1.3 of this QAPP explains the methods used for collecting analytical soil samples during monitoring well installation. Samples will be analyzed for TCL organics, TAL organics, and total organic carbon. The wells to be samples are listed in Table 5-2 of the SAP.

1.2.2.2 Groundwater Samples Collected During Drilling

As described in Section 1.2.1.2, groundwater samples will be collected at 10-foot intervals during drilling at selected monitoring well locations in both source and groundwater investigations. For the groundwater investigation, samples will be collected at MW112, MW113, and MW114. These samples will be collected to determine the maximum depth of contamination, in contrast to the samples collected during the source investigation, which will be collected to determine the specific depths of peak contaminant concentrations. Samples

will be analyzed for VOCs on 24-hour turnaround to allow the field geologist to determine the deepest contaminated interval for placement of the well screen.

1.2.2.3 Groundwater Samples From Monitoring Wells

Groundwater samples for the groundwater investigation will be collected from the 19 Phase II groundwater investigation wells (including two wells installed by the U.S. Geological Survey from October 1992 to February 1993 [MW101D and MW103D]), from 33 Phase I wells, from 21 previously installed Illinois State Water Survey (ISWS) wells, and from 19 industrial wells. All of these samples will be analyzed for VOCs. Because groundwater is currently used as a potable water source in the area, lower detection limits will be required for VOCs to allow a comparison with drinking water standards. Selected wells (20% of all wells to be sampled; the selected wells will be representative of the contaminant plumes in the various aquifers) will be analyzed for general water chemistry parameters including minerals (alkalinity, fluoride, chloride, sulfate, and silica) and nutrients (COD ammonia, total kjeldahl nitrogen, nitrate, nitrite, total phosphorous, and TOC), as well as total dissolved solids and total suspended solids, for remedial design purposes. These parameters are useful in evaluating most conventional remedial treatment methods for contaminated groundwater, including air stripping, carbon adsorption, bioremediation, and chemical treatment methods.

1.2.3 RESIDENTIAL WELL SAMPLING

The residential wells to be sampled in Phase II are a subset of those previously sampled during the Operable Unit study during 1990. The wells to be sampled are those that were located outside of the area where municipal water connections were determined to be necessary. Based on the Operable Unit study, residences within the area defined as exceeding MCLs plus a buffer zone were connected to municipal water supplies. The objective of Phase II residential well sampling is to determine whether the contaminant plume has expanded beyond the previous boundaries defined by the Operable Unit data to areas

underlying any of the residences that were not connected. Twenty-five residential wells will be sampled, as listed in Table 5-4 of the SAP. Samples will be analyzed for VOCs at low detection levels to allow for comparison with drinking water standards.

1.2.4 RESIDENTIAL AIR SAMPLING

Based on the Phase I data, there exists the potential for VOC vapors to migrate upward from the upper part of the saturated zone, through the vadose zone and into confined residential spaces, particularly basements. Therefore, air monitoring will be performed in 14 residential basements, 12 of them located near contaminant source areas. The data from the analysis may be used in risk assessment.

The overall goal of the air sampling program is to establish the extent of exposure to volatile organics for the residents living near contaminant source areas. To accomplish this goal, the soil gas migration pathway will be characterized and the routes of exposure will be identified.

1.2.4.1 Indoor Air Sampling

In the selected area for residential air sampling, 14 residences will be sampled. Twelve of these will be from locations hydraulically downgradient from nearby contaminant source areas, and will be chosen from clusters of five by the IEPA Community Relations team, based on their capabilities to obtain permission from residents. Two additional background residences will be selected in an area where groundwater contamination is known to be negligible.

For each of the residential locations selected, it is proposed that two air samples be collected, one from the breathing zone in the basement and one of ambient air collected adjacent to the residence. The samples will be collected over a 24-hour period using a Summa canister.

One of the objectives of the air quality survey is to determine if residents may be exposed to VOCs from either the soil gas pathway or from ambient air influx from outdoors to indoors.

The air samples from the basement of each residence will be analyzed for volatile organics using EPA Method TO-14. The exact location of the samples will be determined as outlined in Section 3.6.4.1 of the Work Plan.

1.2.4.2 Ambient Air Sampling

An ambient air sample from outside each residence will be collected simultaneously with the indoor air sample to determine the VOC concentrations immediately outside the residence. As described in the SAP, both the ambient air and indoor air samples will be collected over a 24-hour period with a 15-liter Summa canister. The analysis to be performed on the air samples will include volatile organics by EPA Method TO-14 as described in the SAP.

1.3 SAMPLING NETWORK DESIGN AND RATIONALE

The scope of sampling for the Phase II Remedial Investigation includes 131 subsurface soil samples collected during drilling of soil borings and monitoring wells and excavation of test pits; groundwater samples from 31 Phase II source investigation wells; 92 groundwater samples from 33 Phase I wells, 19 Phase II groundwater investigation wells, 21 ISWS wells and 19 industrial wells; 80 groundwater samples collected during drilling (for vertical profiling); groundwater samples from 25 residential wells; 20 air samples from adjacent to test pits; and 28 air samples from residences. Table 1-1 is a summary of the sampling and analysis network and specifies the sample matrix, the parameters to be measured, the number of samples to be collected, and the level of QC effort for each sample type.

All sampling and testing will conform to guidelines set forth in the User's Guide to the EPA Contract Laboratory Program. The QAPP and sections of the SAP discuss the specific

TABLE 1-1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples[1]	QC Samples: Field Duplicates	QC Samples: Field Blank	Matrix Total
Subsurface soil samples collected during drilling of borings and monitoring wells	Qualitative organic vapor screening with Hnu or OVA	CLP RAS Volatile Organics[2,5]	123	13	-	136
		CLP RAS B/N/A Extractable Compounds[2,5]	123	13	-	136
		CLP RAS Pesticides/PCB's[2,5]	123	13	-	136
		CLP RAS Metals[3,5]	123	13	-	136
		CLP RAS Cyanide[5]	123	13	-	136
		CLP SAS for TOC[5]	30	3	-	33
	None	Geotechnical parameters: grain-size analysis	5	1	-	6
		Geotechnical parameters: falling-head permeability	20	-	-	20
Subsurface soils collected during excavation of test pits	Qualitative organic vapor screening with HNu or OVA	CLP High-Concentration Organics	8	1	-	9
		CLP High-Concentration Inorganics	8	1	-	9
		CLP TCLP Parameters	4	1	-	5

1. Frequency of collection for all investigative and QC samples is 1.
2. Contract Laboratory Program Routine Analytical Services (CLP RAS) volatile, extractable and pesticide/PCB compounds are listed in Tables 3-1 through 3-3 of the QAPP.
3. Contract Laboratory Program Routine Analytical Services (CLP RAS) metals parameters are listed in Table 3-4 of the QAPP.
4. CLP SAS parameters for volatile organics are listed in the SAS request forms in Appendix B of the QAPP.
5. No extra sample volume is required for soil matrix spike/matrix spike duplicate (MS/MSD) samples.
6. Samples collected for MS/MSD analysis will be collected at double the volume.
7. Performance evaluation samples will be shipped at a frequency of one per group of 20 or fewer samples.
8. One trip blank will be shipped with each shipment of volatile organics.
9. MS/MSD's will be collected at a frequency of one per group of 20 or fewer samples.

(continued)

TABLE 1-1 (continued)

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples	QC Samples: Field Duplicates	QC Samples: Field Blank	Matrix Total
Groundwater samples from 31 Phase II source investigation wells	pH, temperature, conductivity	Drinking Water Level Analysis for Volatile Organics from CLP SAS[4,7,8]	31	4	4	39
		CLP RAS Pesticide PCBs[2,6,9]	31	4	4	39
		CLP RAS Compounds B/N/A Extractable[2,6,9]	31	4	4	39
		CLP RAS Metals (filtered)[3]	31	4	4	39
		CLP RAS Cyanide (unfiltered)	31	4	4	39
Groundwater samples from 33 Phase I wells, 19 Phase II groundwater investigation wells, 21 ISWS wells, and 19 industrial wells.	pH, temperature, conductivity	Drinking Water Level Analysis for Volatile Organics[4,7,8]	92	10	10	112
Groundwater samples from 25 of the above-noted wells.	pH, temperature, conductivity	CLP SAS analysis for TDS/TSS	25	3	3	31

1. Frequency of collection for all investigative and QC samples is 1.
2. Contract Laboratory Program Routine Analytical Services (CLP RAS) volatile, semi-volatile and pesticide/PCB compounds are listed in Tables 3-1 through 3-3 of the QAPP.
3. Contract Laboratory Program Routine Analytical Services (CLP RAS) metals parameters are listed in Table 3-4 of the QAPP.
4. CLPS SAS parameters for volatile organics are listed in the SAS request forms in Appendix B of the QAPP.
5. No extra sample volume is required for soil matrix spike/matrix spike duplicate (MS/MSD) samples.
6. Samples collected for MS/MSD analysis will be collected at double the volume.
7. Performance evaluation samples will be shipped at a frequency of one per group of 20 or fewer samples.
8. One trip blank will be shipped with each shipment of volatile organics.
9. MS/MSD's will be collected at a frequency of one per group of 20 or fewer samples.

(continued)

TABLE 1-1 (continued)

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Samples	QC Samples: Field Duplicates	QC Samples: Field Blank	Matrix Total
		CLP SAS analysis for minerals (conductivity, alkalinity, fluoride, chloride, sulfate, silica)	25	3	3	31
		CLP SAS analysis for nutrients (ammonia, COD, total Kjeldahl nitrogen, nitrate, nitrite, TOC, total phosporus)	25	3	3	31
Groundwater samples from 25 residential wells.	pH, temperature, conductivity	Drinking Water Level Analysis for Volatile Organics[4,7,8]	25	3	3	31
Groundwater samples collected during drilling, for screening (vertical profiling) purposes.	pH, temperature, conductivity	Fast-turnaround Analysis for halogenated VOCs	80	8	8	96
Air samples from 14 residential basements (including 2 background samples; inside and outside at each location).	None	EPA method TO-14 for VOCs	28	3	3	34
Air samples adjacent to test pits.	None	EPA method TO-1 for VOCs	20	2	2	24

1. Frequency of collection for all investigative and QC samples is 1.
2. Contract Laboratory Program Routine Analytical Services (CLP RAS) volatile, semi-volatile and pesticide/PCB compounds are listed in Tables 3-1 through 3-3 of the QAPP.
3. Contract Laboratory Program Routine Analytical Services (CLP RAS) metals parameters are listed in Table 3-4 of the QAPP.
4. CLPS SAS parameters for volatile organics are listed in the SAS request forms in Appendix B of the QAPP.
5. No extra sample volume is required for soil matrix spike/matrix spike duplicate (MS/MSD) samples.
6. Samples collected for MS/MSD analysis will be collected at double the volume.
7. Performance evaluation samples will be shipped at a frequency of one per group of 20 or fewer samples
8. One trip blank will be shipped with each shipment of volatile organics.
9. MS/MSD's will be collected at a frequency of one per group of 20 or fewer samples.

sampling and analytical procedures to be followed for this project. Sampling network design and rationale are discussed specifically in subsections 4.1 and 4.2 of the SAP and in subsections 3.5 through 3.6 of the Work Plan.

1.4 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are qualitative and quantitative statements which specify the quality of the data required to support decisions made during RI/FS activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels which address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to ARARs, initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). These types of data include those generated on-site through the use of HNu, pH, conductivity, and other real-time monitoring equipment at the site.

- Field Analyses (DQO Level 2): This provides rapid results and better quality than in Level 1. This level may include mobile lab-generated data depending on the level of quality control exercised. Level 2 data will not be generated during the portion of Phase II governed by this QAPP.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile lab-generated data and some analytical lab methods (e.g., laboratory data with quick turnaround used for screening but without full quality control documentation). Level

3 data will be generated from the collection of groundwater samples during drilling, analyzed for VOCs (see Table 1-2).

- Confirmational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives, and PRP determination. These analyses require full Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with EPA recognized protocol. Level 4 data will be generated from the collection of soil and groundwater samples from soil borings and monitoring wells, analyzed for TCL organics and TAL inorganics (see Table 1-2).

- Non-Standard (DQO Level 5): This refers to analyses by non-standard protocols, for example, when exacting detection limits or analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data. Level 5 data will be generated during the collection of soil samples from monitoring wells and test pits, groundwater samples from monitoring and residential wells, and air samples from residences (see Table 1-2).

1.5 SCHEDULE

The anticipated schedule for key activities in this Phase II Remedial Investigation is shown in Figure 1-2.

TABLE 1-2
SOUTHEAST ROCKFORD PHASE II
PRELIMINARY DATA QUALITY OBJECTIVES SUMMARY

DATA GATHERING ACTIVITY	SOIL GAS SURVEY	SOIL BORING INSTALLATION	GROUNDWATER SAMPLING DURING DRILLING	SOIL SAMPLING DURING DRILLING
Objectives and Data uses	Identify contaminated areas during source investigation Evaluate need for additional study/immediate remedial action Provide source information for groundwater modeling of contaminant fate and transport	Confirm and further define nature of contamination in "hot spots" identified in soil gas survey Evaluate need for additional study/immediate remedial action Provide source information for groundwater modeling of contaminant fate and transport	Determine optimal interval to set well screens in areas where the pattern of groundwater contamination with depth is unknown	Define soil contamination near potential source areas Determine background soil chemical characteristics Provide information about contaminant partitioning Provide information for groundwater modeling of contaminant fate and transport and for evaluation of remedial options
Appropriate Analytical Levels	Screening level site evaluation: Level 2	Site characterization: Level 4	Intermediate site characterization: Level 3	Site characterization: Levels 4 and 5:
Data Needs	Target VOCs TCE, TCA, and PCE in potential source areas to target the soil boring program	CLP TCL organics and CLP TAL inorganics to evaluate areas of likely high VOC concentrations	VOCs by GC 24-hour turnaround to identify aquifer intervals with relatively higher concentrations of VOCs	CLP TCL organics and CLP TAL inorganics to evaluate nature and extent of soil contamination and background soil characteristics CLP SAS for TOC to evaluate contaminant partitioning potential

US4264

TABLE 1-2 (Continued)
SOUTHEAST ROCKFORD PHASE II
PRELIMINARY DATA QUALITY OBJECTIVES SUMMARY

DATA GATHERING ACTIVITY	GROUNDWATER SAMPLING	RESIDENTIAL WELL SAMPLING	RESIDENTIAL AIR SAMPLING
Objectives and Data uses	Determine groundwater quality to evaluate nature and extent of contamination potential migration pathways and remedial options Provide data for groundwater modeling of contaminant fate and transport Provide data to evaluate health and environmental risks	Determine drinking water quality to evaluate health risks to residents	Evaluate volatilization from groundwater plume through vadose zone to confined spaces as an exposure pathway Provide data to evaluate health risks associated with the contaminants
Appropriate Analytical Levels	Site characterization: Levels 4 and 5 Risk assessment: Levels 4 and 5	Risk assessment: Level 5	Site characterization: Level 5 Risk assessment: Level 5
Data Needs	CLP SAS for low concentrations VOCs for all wells for extent of contamination CLP TCL BN/A extractables and CLP TAL inorganics for source investigation wells to evaluate nature and extent of contamination CLP SAS for general water quality parameters for selected wells to evaluate treatment techniques	CLP SAS for low concentrations VOCs extent of VOC contamination at drinking water levels	VOCs in air using EPA Method TO-14 to evaluate extent to exposure to residents

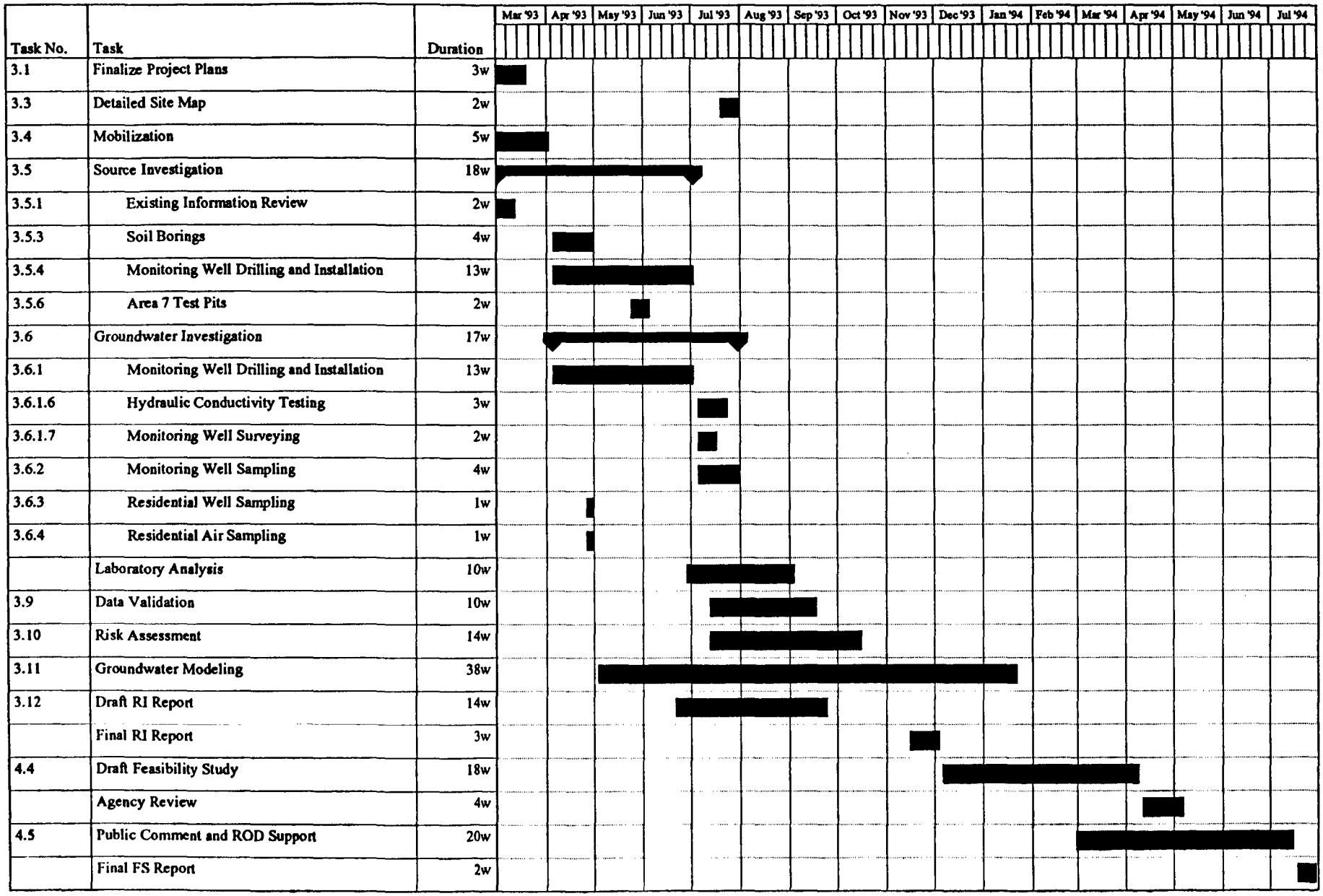
US4265

TABLE 1-2 (Continued)
SOUTHEAST ROCKFORD PHASE II
PRELIMINARY DATA QUALITY OBJECTIVES SUMMARY

DATA GATHERING ACTIVITY	TEST PIT EXCAVATION AND AIR MONITORING
Objectives and Data Uses	Determine nature and extent of wastes in suspected source areas. Provide data to evaluate air quality impacts associated with test pit excavation.
Appropriate Analytical Levels	Site characterization: Level 5 Risk Assessment: Level 5
Data Needs	CLP *High-concentration Organics* and *High-concentration* Inorganics to evaluate extent of waste in suspected source areas TCLP Organics and Inorganics to evaluate leachability and mobility of waste EPA method TO-1 to evaluate air quality impacts

US4266

Figure 1-2
SOUTHEAST ROCKFORD PHASE II RI/FS SCHEDULE



Task No.	Task	Duration
3.1	Finalize Project Plans	3w
3.3	Detailed Site Map	2w
3.4	Mobilization	5w
3.5	Source Investigation	18w
3.5.1	Existing Information Review	2w
3.5.3	Soil Borings	4w
3.5.4	Monitoring Well Drilling and Installation	13w
3.5.6	Area 7 Test Pits	2w
3.6	Groundwater Investigation	17w
3.6.1	Monitoring Well Drilling and Installation	13w
3.6.1.6	Hydraulic Conductivity Testing	3w
3.6.1.7	Monitoring Well Surveying	2w
3.6.2	Monitoring Well Sampling	4w
3.6.3	Residential Well Sampling	1w
3.6.4	Residential Air Sampling	1w
	Laboratory Analysis	10w
3.9	Data Validation	10w
3.10	Risk Assessment	14w
3.11	Groundwater Modeling	38w
3.12	Draft RI Report	14w
	Final RI Report	3w
4.4	Draft Feasibility Study	18w
	Agency Review	4w
4.5	Public Comment and ROD Support	20w
	Final FS Report	2w

Project: Southeast Rockford
Date: 3/8/93

Summary Task

Task

US4267

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

Camp Dresser and McKee Inc. (CDM), as prime contractor, has overall responsibility for all phases of the Southeast Rockford Phase II Remedial Investigation and will oversee the field investigations and prepare the Remedial Investigation report. CDM will also provide QA/QC for all deliverables and provide for their issuance.

2.1 PROJECT ORGANIZATION

The project organization structure (see Figure 2-1) shows the staff designations, assignments and lines of communication for the Phase II Remedial Investigation.

2.2 IEPA PERSONNEL

2.2.1 PROJECT MANAGER

Project Manager, Mr. Paul Takacs, is responsible for overall management and coordination of technical and fiscal aspects of the Phase II Remedial Investigation. Mr. Takacs will be the IEPA contact with the USEPA Region V Project Manager.

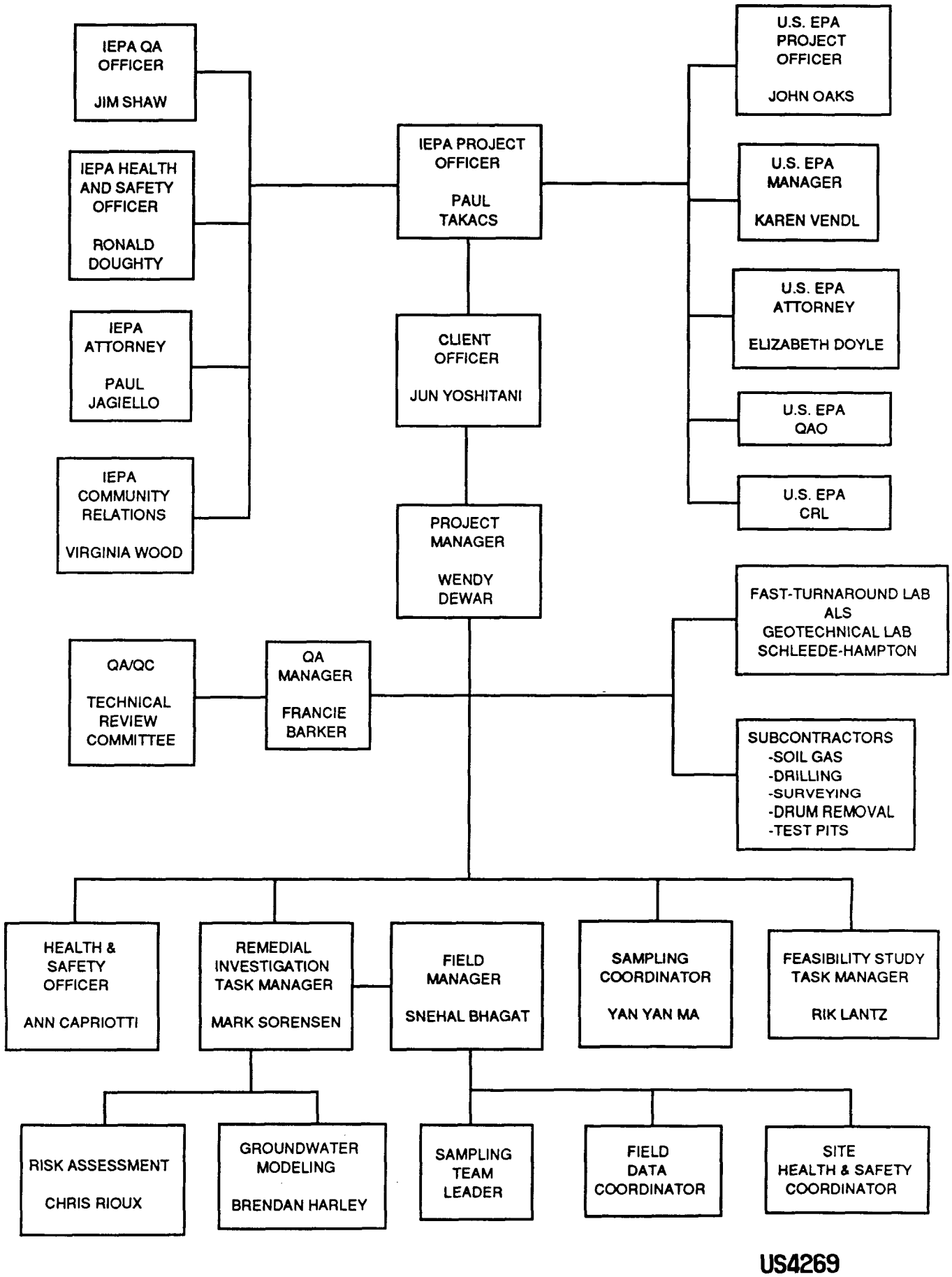
2.2.2 ATTORNEY

The Attorney, Mr. Paul Jagiello, is responsible for the legal aspects of the Phase II Remedial Investigation.

2.2.3 QUALITY ASSURANCE OFFICER

The Quality Assurance Section Project Officer, Mr. Jim Shaw, is responsible for the QAPP technical review. Mr. Shaw is available for consultation on various QA/QC issues.

FIGURE 2-1
SOUTHEAST ROCKFORD PHASE II RI
ORGANIZATION CHART



US4269

2.2.4 HEALTH AND SAFETY OFFICER

The Health and Safety Officer, Mr. Ronald Doughty, is responsible for the review of the Health and Safety Plan.

2.3 USEPA PERSONNEL

2.3.1 STATE PROJECT OFFICER

The USEPA State Project Officer, Mr. John Oakes, is responsible for federal oversight of state-lead activities for the state of Illinois.

2.3.2 REMEDIAL PROJECT MANAGER

The USEPA Region V Remedial Project Manager, Ms. Karen Vendl, is responsible for oversight of the Phase II Remedial Investigation at Southeast Rockford.

2.3.3 QUALITY ASSURANCE SECTION

The USEPA Region V Quality Assurance Section (MQAB/ESD) is responsible for review and approval of the QAPP.

2.3.4 LABORATORY TESTING ASSIGNMENTS

- Analytical Laboratory Services (ALS) of Rockford, Illinois will be contracted to CDM to provide 24-hour turnaround analysis of halogenated volatiles in screening-level groundwater samples collected during drilling, by EPA method 8010.

2.2.4 HEALTH AND SAFETY OFFICER

The Health and Safety Officer, Mr. Ronald Doughty, is responsible for the review of the Health and Safety Plan.

2.3 USEPA PERSONNEL

2.3.1 STATE PROJECT OFFICER

The USEPA State Project Officer, Mr. John Oakes, is responsible for federal oversight of state-lead activities for the state of Illinois.

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The USEPA Region V Remedial Project Manager, Ms. Karen Vendl, is responsible for oversight of the Phase II Remedial Investigation at Southeast Rockford.

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The USEPA Region V Quality Assurance Section (MQAB/ESD) is responsible for review and approval of the QAPP.

2.3.4 LABORATORY TESTING ASSIGNMENTS

- Analytical Laboratory Services (ALS) of Rockford, Illinois will be contracted to CDM to provide 24-hour turnaround analysis of halogenated volatiles in screening-level groundwater samples collected during drilling, by EPA method 8010.

- USEPA Contract Laboratory Program (CLP) will analyze all liquid and soil samples except for screening-level samples as part of the Routine Analytical Services and/or Special Analytical Services packages.

2.3.5 LABORATORY QA/QC RESPONSIBILITIES

- ALS screening-level fast-turnaround samples
  - Requests initiated by CDM sampling team
  - QA/QC procedures described in Appendix F
  - Final data review by CDM Project Organization

- CLP Routine Analytical Services (RAS)
  - Request initiated by CDM sampling team
  - Support Services Branch, Office of Emergency and Remedial Response, USEPA Headquarters
  - USEPA EMSL, Las Vegas
  - Final data review by CDM Project Organization
  - Review of tentatively identified compounds and assessment of need for confirmation by CDM Project Organization

- CLP Special Analytical Services (SAS)
  - Requests initiated by CDM Project Organization
  - Requests coordinated through USEPA Region V Environmental Services Division or USEPA Region V Remedial Response Branch of USEPA Remedial Project Manager (RPM)
  - Review of SAS specifications - USEPA Region V QA Office
  - Final data review by CDM Project Organization

2.3.6 REGIONAL SAMPLE CONTROL COORDINATOR

The USEPA Region V Regional Sample Control Coordinator (RSCC) will be the contact point for the scheduling of CLP SAS analyses. The RSCC will be responsible for training the CDM Field Team Leader in the use of the USEPA CLP and its associated paperwork.

2.4 CONTRACTOR PERSONNEL

CDM, as contractor to IEPA, will validate and analyze the data generated by the Phase II Remedial Investigation field activities. CDM will be responsible for completion of tasks specified in the Statement of Work which includes field measurements, sample collection, and the preparation of the Phase II RI reports.

2.4.1 PROJECT MANAGER

The Project Manager is responsible for day-to-day management and coordination of the contractor staff. This duty includes, but is not limited to, ensuring that all contractor and subcontractor staff understand and comply with the QA/QC program. The Project Manager is responsible for the Work Plan and review of data generated from field measurements and activities. The Project Manager will also be responsible for preparing the Phase II reports.

2.4.2 PROJECT QUALITY ASSURANCE MANAGER

The CDM Quality Assurance Manager is responsible for providing specific QA support to the Project Manager and coordinating QA technical operations among task teams performing duties that are assigned to CDM during this Phase II Remedial Investigation.

2.4.3 FEASIBILITY STUDY MANAGER

The Feasibility Study Manager is responsible for day-to-day management and coordination of the contractor staff for the purposes of completing the tasks necessary for the Feasibility Study. The Feasibility Study Manager will be responsible for directing the preparation of the FS report.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results which are legally defensible in a court of law. Specific procedures to be used for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

3.1 REGULATORY AND LEGAL REQUIREMENTS

The data obtained from analysis of the production and residential wells will be compared to the National Primary Drinking Water Standards. The method detection limits for analytical services from the CLP are sufficiently low to allow this comparison for the compounds of concern. The method detection limits for CLP Analysis are presented in Section 3.3.

3.2 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, duplicate and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks consist of distilled water, will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the site which may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement

methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One matrix spike/matrix spike duplicate will be collected for every 20 or fewer investigative samples. MS/MSD samples are designated/collected for organic analyses being sent to the CLP only.

The general level of the QC effort will be one field duplicate and one field blank for every 10 or fewer investigative samples. One volatile organic analysis (VOA) trip blank consisting of distilled deionized ultra-pure water will be included along with each shipment of aqueous VOA samples.

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil). The number of duplicate and field blank samples to be collected are listed in Table 1-1. Sampling procedures are specified in the Sampling and Analysis Plan.

Performance Evaluation Samples (PES) will be submitted with the low concentration volatile organic aqueous samples. One PES will be submitted for every 20 samples shipped or once during each 7-day calendar period of sample shipping, whichever comes first. PES samples will be supplied to CDM by USEPA. Acceptance criteria for PES samples will be as per Method 6/91 in the drinking water SAS in Appendix B.

The specific level of field QC effort for the Southeast Rockford Phase II Remedial Investigation is described in Section 3.0 of the Sampling and Analysis Plan (SAP) and is summarized by sample matrix and parameter in Table 1-1 of this QAPP.

Groundwater samples collected during drilling will be sent to a local laboratory (ALS) for analysis of halogenated organics for vertical profiling purposes (Analytical Level III). The

level of laboratory QC effort for these samples is specified in the laboratory's QA/QC manual (Appendix E).

All other soil, groundwater, air, and residential well samples will be sent to the CLP Laboratory for analysis. The analysis will be according to both RAS and SAS protocols (Analytical Levels IV and V) for organics and inorganics. The level of laboratory QC effort for RAS analysis provided by the CLP is specified in the current statements of work (SOW/OLM01.1 for organic and SOW/ILM01.0 for inorganic analyses for CLP, low to medium concentration; SOW 9/88 for High-concentration organics and SOW/IHC01.1 for High-concentration inorganics for CLP). Tables 3-1, 3-2, 3-3, and 3-4 contain the quantitation levels for organic and inorganic compounds (low and medium concentrations), respectively. The CLP will analyze certain soil, air, and groundwater samples under the provisions of special analytical services, as well as samples collected from residential wells. The level of Laboratory QC effort for special analytical services (SAS) analyses is outlined individually in each SAS request contained in Appendix B.

3.3 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSIS

The fundamental QA objective with respect to accuracy, precision, and sensitivity (see glossary of terms for definitions, Section 15.0) of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. The accuracy and precision requirements for CLP RAS analysis are specified in the current SOW OLM01.0 (8/91) for organic analysis (low-medium concentration) and the current SOW ILM02.0 (9/91) for inorganic analysis (low-medium concentration); accuracy and precision requirements are specified in the current SOW (9/88) for CLP High-concentration organic analyses and the current SOW IHC01.1 for CLP High-concentration inorganics. The sensitivities required for CLP analyses will be the method detection limits shown in Tables 3-1 through 3-4, from the cited SOWs. The accuracy and precision requirements for SAS from the CLP are specified in the SAS request forms (Appendix B). The sensitivities required for SAS CLP analyses are given for each

TABLE 3-1

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Volatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
1. Chloromethane	74-87-3	10	10	1200	(50)
2. Bromomethane	74-83-9	10	10	1200	(50)
3. Vinyl Chloride	75-01-4	10	10	1200	(50)
4. Chloroethane	75-00-3	10	10	1200	(50)
5. Methylene Chloride	75-09-2	10	10	1200	(50)
6. Acetone	67-64-1	10	10	1200	(50)
7. Carbon Disulfide	75-15-0	10	10	1200	(50)
8. 1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9. 1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10. 1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11. Chloroform	67-66-3	10	10	1200	(50)
12. 1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13. 2-Butanone	78-93-3	10	10	1200	(50)
14. 1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15. Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16. Bromodichloromethane	75-27-4	10	10	1200	(50)
17. 1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18. cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19. Trichloroethene	79-01-6	10	10	1200	(50)
20. Dibromochloromethane	124-48-1	10	10	1200	(50)
21. 1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22. Benzene	71-43-2	10	10	1200	(50)
23. trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24. Bromoform	75-25-2	10	10	1200	(50)
25. 4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26. 2-Hexanone	591-78-6	10	10	1200	(50)
27. Tetrachloroethene	127-18-4	10	10	1200	(50)
28. Toluene	108-88-3	10	10	1200	(50)
29. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
30. Chlorobenzene	108-90-7	10	10	1200	(50)
31. Ethyl Benzene	100-41-4	10	10	1200	(50)
32. Styrene	100-42-5	10	10	1200	(50)
33. Xylenes (Total)	1330-20-7	10	10	1200	(50)

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher

TABLE 3-2
(continued)

Semivolatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
69. Dibenzofuran	132-64-9	10	330	10000	(20)
70. 2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71. Diethylphthalate	84-66-2	10	330	10000	(20)
72. 4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73. Fluorene	86-73-7	10	330	10000	(20)
74. 4-Nitroaniline	100-01-6	25	800	25000	(50)
75. 4,6-Dinitro-2-methylphenol	534-52-1	25	800	25000	(50)
76. N-nitrosodiphenylamine	86-30-6	10	330	10000	(20)
77. 4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
78. Hexachlorobenzene	118-74-1	10	330	10000	(20)
79. Pentachlorophenol	87-86-5	25	800	25000	(50)
80. Phenanthrene	85-01-8	10	330	10000	(20)
81. Anthracene	120-12-7	10	330	10000	(20)
82. Carbazole	86-74-8	10	330	10000	(20)
83. Di-n-butylphthalate	84-74-2	10	330	10000	(20)
84. Fluoranthene	206-44-0	10	330	10000	(20)
85. Pyrene	129-00-0	10	330	10000	(20)
86. Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87. 3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88. Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89. Chrysene	218-01-9	10	330	10000	(20)
90. bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	(20)
91. Di-n-octylphthalate	117-84-0	10	330	10000	(20)
92. Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93. Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
94. Benzo(a)pyrene	50-32-8	10	330	10000	(20)
95. Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	(20)
96. Dibenz(a,h)anthracene	53-70-3	10	330	10000	(20)
97. Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

TABLE 3-3

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Pesticides/Aroclors	CAS Number	Quantitation Limits* Water ug/L	Soil ug/Kg	On Column (pg)
98. alpha-BHC	319-84-6	0.05	1.7	5
99. beta-BHC	319-85-7	0.05	1.7	5
100. delta-BHC	319-86-8	0.05	1.7	5
101. gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
102. Heptachlor	76-44-8	0.05	1.7	5
103. Aldrin	309-00-2	0.05	1.7	5
104. Heptachlor epoxide	1024-57-3	0.05	1.7	5
105. Endosulfan I	959-98-8	0.05	1.7	5
106. Dieldrin	60-57-1	0.10	3.3	10
107. 4,4'-DDE	72-55-9	0.10	3.3	10
108. Endrin	72-20-8	0.10	3.3	10
109. Endosulfan II	33213-65-9	0.10	3.3	10
110. 4,4'-DDD	72-54-8	0.10	3.3	10
111. Endosulfan sulfate	1031-07-8	0.10	3.3	10
112. 4,4'-DDT	50-29-3	0.10	3.3	10
113. Methoxychlor	72-43-5	0.50	17.0	50
114. Endrin ketone	53494-70-5	0.10	3.3	10
115. Endrin aldehyde	7421-36-3	0.10	3.3	10
116. alpha-Chlordane	5103-71-9	0.05	1.7	5
117. gamma-Chlordane	5103-74-2	0.05	1.7	5
118. Toxaphene	8001-35-2	5.0	170.0	500
119. Aroclor-1016	12674-11-2	1.0	33.0	100
120. Aroclor-1221	11104-28-2	2.0	67.0	200
121. Aroclor-1232	11141-16-5	1.0	33.0	100
122. Aroclor-1242	53469-21-9	1.0	33.0	100
123. Aroclor-1248	12672-29-6	1.0	33.0	100
124. Aroclor-1254	11097-69-1	1.0	33.0	100
125. Aroclor-1260	11096-82-5	1.0	33.0	100

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of Pesticides/Aroclors.

TABLE 3-4

INORGANIC TARGET ANALYTE LIST (TAL)

Analyte	Contract Required Detection Limit (1,2) (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

(1) Subject to the restrictions specified in the first page of Part G, Section IV of Exhibit D (Alternate Methods - Catastrophic Failure) any analytical method specified in SOW Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Detection Limit. This is illustrated in the example below:

For lead:

Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 220
Contract Required Detection Limit (CRDL) = 3

TABLE 3-4

(continued)

The value of 220 may be reported even though the instrument detection limit is greater than CRDL. The instrument or method detection limit must be documented as described in Exhibits B and E.

(2) The CRDLs are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix.

compound in the SAS request forms. The accuracy, precision and sensitivity requirements for the screening levels samples sent to ALS are provided in Appendix E.

Data from field measurements will be assessed by thorough review of QC data (calibrations, standards, blanks, and replicates as discussed in Section 3.5), documentation that analytical procedures were adhered to, and reports from system audits.

3.4 COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

It is expected that the CLP will provide data meeting QC acceptance criteria for 95 percent or more of all samples tested. Analytical data from the CLP is assessed for contractual completeness by the Sample Management Office according to their contract compliance screening procedure before the data is sent to the CDM project organization. The data is then reviewed for precision, accuracy, and completeness in accordance with the procedures described in the National Functional Guidelines for Organic Data Review published by the USEPA Contract Laboratory Program in Draft form, June 1991; in Laboratory Data Validation: Functional Guidelines for Evaluating Inorganics Analyses published by the USEPA Data Review Work Group July 1988; or as outlined in the SAS. Completely valid data are required for samples in the SAP as "background samples". The CLP laboratory should provide data that are complete and valid. The screening level volatile organic data will be reviewed in the field and is expected to provide data that is 90% complete. All field data will be reviewed for completeness by the principal investigator. (For definitions of completeness, representativeness and comparability, see Section 15, Glossary of Terms). This Phase II investigation has been designed to provide sufficient data to achieve the project objectives (subsection 1.2).

The sampling network was designed to provide data representative of site conditions. To achieve the goal of obtaining representative site data the standard operating procedures described in this QAPP and SAP will be adhered to during the project. During development of this sampling network, consideration was given to past waste disposal practices, existing

analytical data, physical setting and processes. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in this QAPP, are expected to provide data that will be comparable to the data collected in the operable unit study and in the Phase I study of this investigation. This will be achieved by using CLP RAS/SAS analytical protocols, QA/QC, and reporting. These new analytical data, however, may not be directly comparable to all existing data (USEPA TAT and IDPH) because of differences in procedures and QA objectives. Representativeness of Phase II data will be assessed by comparison of field duplicates results and by comparison of Phase II data with Phase I and Operable Unit data where applicable.

3.5 FIELD MEASUREMENTS

Measurement data will be generated in many field activities that are incidental to collecting samples for analytical testing or unrelated to sampling. These activities include, but are not limited to, the following:

- Documenting time and weather conditions;
- Locating and determining the elevation of sampling stations;
- Determining Ph and temperature of water supply;
- Determining depths in a borehole or well;
- Verifying well development and pre-sampling purge volumes;
- Indoor and ambient air sampling; and
- Well sampling.

The general QC objective for such measurement data is to obtain reproducible and comparable measurements to a degree of accuracy consistent with the intended use of the data through the documented use of standardized procedures. The procedures for performing these activities and the standardized formats for documenting them are presented in the SAP.

Completeness, representativeness and comparability (defined in Section 15.0) are discussed below and are addressed in the analytical field procedures (Appendix C). The completeness for analytical field results for this phase will be 90% or better. Representative field data will be obtained by performing all field sampling and field measurements in a standardized manner by strictly adhering to the procedures specified in this QAPP and in the SAP and Work Plan. Qualitative comparability will be achieved by following QA/QC sampling and analytical procedures outlined in the QAPP, SAP and Work Plan.

The precision and accuracy of Ph measurements will be assessed in the field prior to analysis. The calibration of the Ph meter will be conducted at the beginning of the day prior to use. The calibration of the Ph meter (Appendix C) will be performed by taking two measurements on each of two standard buffer solutions of Ph 4 and Ph 7. The accuracy will be determined by the difference in replicate samples of the standard Ph buffer solutions. These measurements should be within $\pm$ 0.1 Ph units from the value of the standard solutions. Replicate analysis will be completed on both standards and the difference between the replicates will be within $\pm$ 0.1 standard Ph units of the known value of the standard buffer solution. The precision will be less than or equal to 0.1 difference between the two measurements on each pH standard buffer solution. If the pH meter fails to calibrate properly, a different pH meter will be calibrated and used.

The calibration measurements made for the specific conductance will be used to assess the accuracy and precision of the instrument used. The calibration of the instrument will be made by making two measurements on a standard. The standard used will be a solution of 0.01 demol KC1. The accuracy will be within 10% of the standard value and precision will be less than or equal to 15% of the difference between the two replicate measurements of the

standard. If the measurements are not within ± 10% of the standard or are not reproducible within ± 15%, the instruments will be returned to the manufacturer for maintenance and calibration.

The calibration procedures for the HNu are outlined in Appendix C. The HNu will be calibrated after each field use or prior to each field use if the instrument has not been calibrated during the previous 14 calendar days. Isobutylene will be used as the standard. The instrument will be calibrated in the 0-20 ppm range and the 20-200 ppm range. If the instrument measurements are not within ± 15% of the known standard in either of the two calibration ranges, the instrument will be sent back to the manufacturer for maintenance and calibration.

The calibration procedures for the Organic Vapor Analyzer (OVA) are outlined in Appendix D. This instrument will be calibrated after each field use or prior to each field use if the instrument has not been calibrated during the previous 14 calendar days. The manufacturer calibrates the OVA with methane at the factory. The minimum detection limit for methane is 0.2 ppm. For precise analysis, it is necessary to recalibrate with the specific compound of interest. A commercially available standard will be used if it is necessary to recalibrate for a specific compound. If the instrument is not within ± 15% of the standard, the instrument will be sent back to the manufacturer to be recalibrated.

The level of QC for the thermometer will consist of a calibration check using an ice/water slurry once at the beginning of field activities. The thermometer must read ± 0.5° C. If the thermometer is out of calibration, it will be replaced.

4.0 SAMPLING PROCEDURES

Procedures to sample groundwater from monitoring wells and to collect soil samples are described in the SAP and in Appendix C. Also included are descriptions of sampling containers, sample preservation techniques, and procedures for sample bottle and sampler decontamination, sample documentation, packaging and shipping. A summary of the sampling and analysis network for this project is provided as Table 1-1. A summary of the sample containers, sample preservation techniques, and holding times are provided in Tables 4-1 and 4-2 for each matrix to be sampled. Standard sampling procedures are referenced and discussed in the Sampling and Analysis Plan.

The Southeast Rockford Phase II Remedial Investigation will use the IEPA Sample Bottle Program. Sample containers will be prepared as specified in the IEPA Exhibit A, Scope of Work for FY90, Sample Bottle Supply Service (Appendix D). The IEPA sample bottle contractors' quality control data, generated for the lots used in this project, will be available upon request. This data may be obtained from the contractor through the IEPA Project Manager.

TABLE 4-1

SAMPLE VOLUMES, CONTAINERS, AND PRESERVATION TECHNIQUES
LOW AND MEDIUM CONCENTRATION

Analysis	Container	Preservative	Maximum Holding Time	Volume of Sample	Matrix
Base/neutral/acid extractables, pesticides/PCBs	Three one-liter amber glass bottles with Teflon lined cap	Cool, 4°C	5 days until extraction. Analyze 40 days after extraction	Fill bottle to neck	Water
Volatiles	Four 40-ml volatile organic analysis (VOA) vials	2 drops concen. HCl to pH<2; cool, 4°C	7 days	Fill completely (no air bubbles	Water
Halogenated Volatiles (for vertical profiling during drilling)	Two 40-ml VOA vials	2 drops concen. HCl; cool, 4°C	7 days	Fill completely (no air bubbles)	Water
Metals (filtered)	One 1-liter high density polyethylene bottle	5-ml 1:1 HNO_3 to pH<2	180 days (28 days for mercury)	Fill to shoulder of bottle	Water
Cyanide (unfiltered)	One 1-liter polyethylene bottle	5-ml 6N NaOH to pH pH>12; cool, 4°C	12 days	Fill to shoulder of bottle	Water

Analysis	Container	Preservative	Maximum Holding Time	Volume of Sample	Matrix
Minerals Alkalinity Fluoride Chloride Sulfate Silica	One one-liter high density polyethylene bottle	Cool, 4°C	14 days	Fill to shoulder of bottle	Water
Nutrients Ammonia COD TKN NO_3-NO_2 TOC Total Phosphorus	One one-liter polyethylene bottle	2-ml 1:1 H_2SO_4 to pH<2; cool, 4°C	28 days	Fill to shoulder of bottle	Water
TDS and TSS	One 1-liter polyethylene bottle	Cool, 4°C	7 days	Fill to shoulder of bottle	Water
Semi-volatiles (extractables and pesticides/PCBs)	One 8-ounce glass wide mouth bottle with Teflon-lined cap	Iced to 4°C	14 days until extraction and analyzed within 40 days after extraction	Fill no more than 3/4 full	Soil
Volatiles	Two 120-ml glass wide-mouth vials with Teflon lined caps	Iced to 4°C	7 days	Fill completely (No Head Space)	Soil
Metals and Cyanide	One 8-ounce glass wide-mouth bottle	Iced to 4°C	180 days (28 days for mercury) and 12 days, respectively	Fill no more than 3/4 full	Soil

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Analysis	Container	Preservative	Maximum Holding Time	Volume of Sample	Matrix
TOC	One 4-ounce glass bottle	Iced to 4°C	28 days	Fill 3/4 full	Soil
Geotechnical (grain-size)	One 8-ounce glass bottle	None	None	Fill 3/4 full	Soil
Geotechnical (falling-head permeability)	One 3-foot long, 2-inch I.D. thin-walled tube (Shelby tube)	None	None	Fill completely as per ASTM method D-1587	Soil
Volatiles	15-liter stainless steel Summa canister	None	30 days	Not applicable	Air
Volatiles	Tenax tube	None	30 days	Not applicable	Air

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TABLE 4-2

SAMPLE VOLUME, CONTAINERS, AND PRESERVATION TECHNIQUES

HIGH CONCENTRATION

Analysis	Container	Preservative	Maximum Holding Time	Volume of Sample
Organics	One 4-oz. wide-mouth glass jar	cool to 4°C	Not established	Fill 1/2 to 3/4 full
Inorganics and Cyanide	One 4-oz. wide-mouth glass jar	cool to 4°C	Not established	Fill 1/2 to 3/4 full
TCLP Organic Parameters	One 1-quart wide-mouth glass jar	cool to 4°C	14 days to extraction; 28 days total	Fill 1/2 to 3/4 full
TCLP Inorganic Parameters	One 1-liter wide-mouth glass jar	cool to 4°C	180 days to extraction (28 days for mercury) 56 days total for mercury	Fill 1/2 to 3/4 full

NOTE:

The normal procedures used for preservation and holding of environmental samples for analysis will not apply to the analysis of high concentration samples. Due to the expected high levels of constituents believed to be present in the samples, losses due to lack of preservation after receipt of the samples from the field are not considered a major problem. If the samples are analyzed within the required time as set forth in the SOW, the samples will not require any preservative prior to preparation, but preservation after preparation and prior to analysis (as described in the individual methods) will be required. The medium level preservation requirements thereby apply. (See Table 3-1.)

US4291

5.0 SAMPLE CUSTODY

5.1 INTRODUCTION

It is USEPA and Region V policy to follow the USEPA Region V sample custody or chain-of-custody protocols as described in "NEIC Policies and Procedures", EPA-330/9-78-001-R, revised May 1986. This Custody is in three parts: sample collection, laboratory, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if the documents:

- are in your possession;
- are in your view, after being in your possession;
- were in your possession and you placed them in a secured location; or
- are in a designated secure area.

5.2 FIELD-SPECIFIC CUSTODY PROCEDURES

The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain-of-custody intact.

Field procedures are as follows:

(a) The field sampler is personally responsible for the care and custody of the

samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

(b) All bottles will be tagged with sample numbers and locations. If applicable, the Sample Management Office (SMO) number and stickers will be affixed.

(c) Sample tags are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint would not function in freezing weather.

(d) The contractor's site manager must review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required. He or she should notify the Project Manager of any breach or irregularity in chain-of-custody procedures.

Transfer of custody and shipment procedures are as follows:

(a) Samples are accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

(b) Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and secured with strapping tape and EPA custody seals for shipment to the

laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

(c) Whenever samples are split with a source or government agency, a separate sample receipt is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "received by" space.

(d) All shipments will be accompanied by the chain-of-custody record identifying the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for return to the sampling office.

(e) If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

5.3 LABORATORY CUSTODY PROCEDURES

Laboratories that are in the CLP as well as non-CLP laboratories authorized to do SAS analyses will follow the sample custody procedures specified in the current CLP SOW OLM01.0 (8/91) for low-medium concentration organic analysis and the current CLP SOW ILM02.0 (9/91) for inorganic low-medium concentration analysis, and in the current CLP

SOW (9/88) for high-concentration organics and the current CLP SOW IHC01.1 for high-concentration inorganics. These custody procedures along with the holding time requirements for CLP samples are described in the appropriate SOW documents. The custody procedures for the screening level laboratory, ALS, are detailed in Appendix E.

5.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

For all samples analyzed under CLP SOWs OLM01.0, OLC01.0, ILC01.0, or ILM02.0, IHC01.1, or SOW 9/88, the CLP laboratories will submit all original documents to USEPA Region V within 35 days of receipt of last sample for the case or SAS contract. The Laboratory Science Support Section (LSSS) will be responsible for the final evidence audit of these files and their secure storage. Any samples analyzed under the 2/88 or 7/88 CLP SOWs will be subjected to an NEIC evidence screen prior to their secure storage as described in the respective SOWs.

The contractor will maintain the site files along with all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and the data and data reviews of the CLP and screening level generated laboratory data in a limited access area and under custody of the contractor's Site Manager.

The final evidence file will include, but not be limited to:

Project Plans	Graphs
Field Data Records	Calculations
Logbooks	Raw Data Summaries
Sample Tags	Data/Purge Files
Chain-of-Custody Records	Correspondence
Sample Tracking Records	Data Validation Files and Reports
Analytical Logbook Pages	Report Notes
Bench Sheets	Miscellaneous-Photos, Maps, Drawings,

Instrument Readout Records	etc.
Computer Printouts	Final Report

EPA United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report & Chain of Custody Record
(For Organic CLP Analysis)

SAS No. (if appl.): NA

Case No.: 17524

1. Project Code: ~~ZF3095~~	Account Code:	2. Region No.: V	Sampling Co.: E&E ARCS	4. Date Shipped: 1/7/92	Carrier: Fed Ex
Regional Information: TFA 102		Sampler (Name): Cathy Kouris		Airbill Number: 3110833880	
Non-Superfund Program		Sampler Signature: Cathy Kouris		5. Ship To: PACE, 1710 Douglas Drive, Minneapolis, MN 55422, Attn: Paul Ernst	
Site Name		3. Type of Activity: Lead / Remedial / Removal; SF, PRP, ST, FED; PA, SSI, LSI; RIFS, RD, RA, O&M, NPLD; CLEM, REMA, REM, OIL, UST			
City, State: Elkhart, IN	Site Spill ID: 7Y				

6. Preservative (Enter in Column D): 1. HCl; 2. HNO3; 3. NaHSO4; 4. H2SO4; 5. Other (SAS) (Specify); 6. Ice only; N. Not preserved

7. Sample Description (Enter in Column A): 1. Surface Water; 2. Ground Water; 3. Leachate; 4. Rinsate; 5. Soil/Sediment; 6. Oil (SAS); 7. Waste (SAS); 8. Other (SAS) (Specify)

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type: Comp / Grab	D Preservative from Box 6	E RAS Analysis: VOA	BNA	Pest/PCB	High ARO/TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Inorg Samp. No.	K Designated Field QC
EPP17	5	L	G	6	X	X	X		130626-28	B20C (5-6.5ft)	1/7/92 805		MEJK06	
EPP18	5	L	G	6	X	X			130629-31	B20E (10-12ft)	1/7/92 830		MEJK07	D-EPP18
EPP19	5	L	G	6	X	X			130632-35	B20E-D (10-12ft)	1/7/92 830		MEJK08	~~Field Duplicate~~
EPP20	5	L	G	6	X	X	X		130636-38	B21 (10-12ft)	1/7/92 915		—	
EPP21	2	L	G	1	X				130639-40	CRMW11S-01	1/7/92 1005		MEJK09	
EPP21	2	L	G	6		X	X		130641-42	CRMW11S-01	1/7/92 1005		MEJK09	D-EPP21
EPP22	2	L	G	1	X				130643-44	CRMW11S-01	1/7/92 1005		MEJK10	~~Field Duplicate~~
EPP22	2	L	G	6		X	X		130645-46	CRMW11S-01	1/7/92 1005		MEJK10	~~Field Duplicate~~ D-
EPP23	4	L	G	1	X				130647-48	CRMWEB-01	1/7/92 1115		MEJK11	~~Field Blank~~ B
EPP23	4	L	G	6		X	X		130649-50	CRMWEB-01	1/7/92 1115		MEJK11	~~Field Blank~~ B

Shipment for Case complete? (Y/N)

Page 1 of 1

Sample used for a spike and/or duplicate: EPP17, EPP21

Additional Sampler Signatures: if this is the sample for MS/MSD, more sample bottles would be collected than this.

Chain of Custody Seal Number: 175654-55

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Cathy Kouris	1/7/92 1830				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)

EPA Form 0-2 (Rev. 8-91) Replaces EPA Form (2075-T), previous edition which may be used

USA237

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ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

230 South Dearborn Street
Chicago, Illinois 6060

93ZF03

CHAIN OF CUSTODY RECORD

PROJ. NO. ~~ZF3035~~ | PROJECT NAME

SAMPLERS: (Signature) Cathy Kouris

Cooler #107

REMARKS: Low Concentration

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	VOA(HCl) 40ml	ABN				TAG Number	MATRIX
501 ~~ZF...~~	1/7/92	805		X	CRMW11S-01+MSD	8	6	2				169170-77	groundwater
502 ~~ZF...~~	1/7/92	825		X	CRMW02S-02	3	3					169178-80	groundwater
502 ~~ZF...~~	1/7/92	825		X	CRMW02S-02 Dup	3	3					169181-83	groundwater
503 ~~ZF...~~	1/7/92	915		X	CRMW23S-01	4	3	1				169184-86	groundwater
201 ~~ZF...~~	1/7/92	1300		X	FB01	4	3	1				169187-89	groundwater

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(CRL TRACKING FORMS IN COOLER)

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Cathy Kouris	1/7/92 1830				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Remarks: Shipped Fed Ex to CRL
Airbill # 1060853196
Custody Seals 163[illegible]-54

Distribution: White – Accompanies Shipment; Pink – Coordinator Field Files; Yellow – Laboratory File

US4298

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Case # or Project Code 1	Sample Number 2	Month/Day/Year 3	Time 4	DESIGNATE Grab 5	DESIGNATE Comp	Station Number and Location 6	Samplers (signatures) 7	PRESERVATIVE: H_2SO_4 ☐ HCL ☐ HNO_3 ☐ NaOH ☐ Other ☐ 8	ANALYSES	
									VOA	METALS
									ABN	CYANIDE
									PEST/PCB 9	
										Mercury
									Pesticides	Fluoride
									Herbicides	Nitrate/Nitrite
									PCB	TOC
									PCDD/PCDF	BOD
									2,3,7,8-TCDD	COD
									Ames Mutagen	TDS
									Asbestos	TSS
									Phosphorus	O&G
									TO1	Sulfate
									TO2	Chloride
										Sulfide
									TOX	Ammonia
									CBOD	Alkalinity
									Bio-Acute	Acidity
									Bio-Chronic	TKN
								Remarks: 10		
								USE FOR MS/MSD ☐ 11		
Tag Number 5— 198823									Lab Sample Number	

Case # or Project Code ZF30035	Sample Number CRMW115-01	Month/Day/Year 1/7/92	Time 1005	DESIGNATE Grab X	DESIGNATE Comp.	Station Number and Location CRMW115-01	Samplers (signatures) Cathy Kovacs	PRESERVATIVE: H_2SO_4 ☐ HCL ☐ HNO_3 ☒ NaOH ☐ Other ☐	ANALYSES	
									VOA	METALS
									ABN	CYANIDE
									PEST/PCB	
										Mercury
									Pesticides	Fluoride
									Herbicides	Nitrate/Nitrite
									PCB	TOC
									PCDD/PCDF	BOD
									2,3,7,8-TCDD	COD
									Ames Mutagen	TDS
									Asbestos	TSS
									Phosphorus	O&G
									TO1	Sulfate
									TO2	Chloride
										Sulfide
									TOX	Ammonia
									CBOD	Alkalinity
									Bio-Acute	Acidity
									Bio-Chronic	TKN
								Remarks: ~~CASE: 17524~~ ~~MEJK 09~~		
								USE FOR MS/MSD ☐		
Tag Number 5— 198824									Lab Sample Num	

Case # CLP Sample #

US4299

EPA Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Special Analytical Services
Packing List/Chain of Custody

69 and E

1. Project Code	Account Code	2. Region No.	Sampling Co.	4. Date Shipped	Carrier
~~ZF3095~~		V	E&E ARCS	1/7/92	Fed Ex

Regional Information: TFA 102
Non-Superfund Program:
Sampler (Name): Cathy Kouris
Sampler Signature: Cathy Kouris
Airbill Number: 3110834053
5. Ship To: CENTEC, 1401 Municipal Rd, Roanoke, VA 24012, ATTN: B Hutchenson
City, State | Site Spill ID

3. Type of Activity — Lead / Remedial / Removal

	Lead	Remedial	Removal
SF	X	RIFS	CLEM
PRP		PA	RD	REMA
ST		SSI	RA	REM
FED		LSI	O&M	OIL
			NPLD	UST

6. Sample Description (Enter In Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil
7. Waste
8. Other (Specify)

7. Preservative (Enter In Column C)
1. HCl
2. HNO_3
3. $NAHSO_4$
4. H_2SO_4
5. NAOH
6. Other (SAS) (Specify)
7. Ice only
N. Not preserved

Sample Numbers	A Matrix Enter from Box 6	B Conc Low Med High	C Preservative Used from Box 7	D Analysis	E Regional Specific Tracking Number or Tag Number	F Station Location Identifier	G Mo/Day/Year/Time Sample Collection	H Sampler Initials	I Designated Field QC
1. E-01	2	L	7	BOD	130759	CRMW025-02	1/7/92 1005		
2. E-01	2	L	4	COD	130760	CRMW025-02	1/7/92 1005		
3. E-02	2	L	7	BOD	130761	CRMW025-02D	1/7/92 1005		~~Field Dup~~
4. E-02	2	L	4	COD	130762	CRMW025-02D	1/7/92 1005		~~Field Dup~~
5. E-03	2	L	7	BOD	130763	CRMW235-01	1/7/92 1115		
6. E-03	2	L	4	COD	130764	CRMW235-01	1/7/92 1115		
7. E-04	2	L	7	BOD	130765	CRMW018-01B	1/7/92 1300		~~Field Blank~~
8. E-04	2	L	4	COD	130766	CRMW018-01B	1/7/92 1300		~~Field Blank~~
9.									
10.									

Shipment for SAS complete? (Y/N): Y
Page 1 of 1
Sample Used for Spike and/or Duplicate: E-03
Additional Sampler Signatures:
Chain of Custody Seal Number: 175672-73

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Cathy Kouris	1/7/92 1830				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples: ☐ Accepted (Signature) ☐ Declined

EPA Form: 9110-3 (7/91)
DISTRIBUTION:

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US4300

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report & Chain of Custody Record

(For Inorganic CLP Analysis)

(If applicable) NA

Case 17524

1. Project Code	Account Code	2. Region No.	Sampling Co.	4. Date Shipped	Carrier
~~ZF3095~~		V	EAERCS	1/7/92	Fed Ex

Regional Information	Sampler (Name)	Airbill Number
TFA 102	Cathy Kouris	3110833891

Non-Superfund Program	Sampler Signature	5. Ship To
	Cathy Kouris	KEYTX 8300 West PARK DR. HOUSTON, TX 77063 ATTN: Dela MASSOUDI

Site Name

City, State | Site Split ID

4. Type of Activity

	Lead	Pre-Remedial	Remedial	Removal
F	X		RIFS X	CLEM
PRP		PA	RD	REMA
ST		SSI	RA	REM
FED		LSI	O&M	OIL
			NPLD	UST

6. Preservative (Enter in Column D)
1. HCl
2. HNO_3
3. NaOH
4. H_2SO_4
5. $K_2CR_2O_7$
6. Ice only
7. Other (SAS) (Specify)
N. Not preserved

7. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type: Comp / Grab	D Preservative from Box 6	E - RAS Analysis: Metals Total	Metals Dissolved	Metals Cyanide	Low Conc. Nitrate/Nitrite	Low Conc. Fluoride	High pH	High Conductivity	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Org. Samp. No.	K Designated Field QC
MEJK06	5	L	G	N	X		X					130709	B20C(5-6.5ft)	1/7/92 805		EPP17	
MEJK07	5	L	G	N	X		X					130710	B20E(10-12ft)	1/7/92 830		EPP18	DUP MEJK07
MEJK08	5	L	G	N	X		X					130711	B20E-D(10-12ft)	1/7/92 830		EPP19	~~Field Duplicate~~
MEJK09	2	L	G	2	X							130712	CRMW115-01	1/7/92 1005		EPP21	
MEJK09	2	L	G	3			X					130713	CRMW115-01	1/7/92 1005		EPP21	DUP MEJK09
MEJK10	2	L	G	2	X							130714	CRMW115-01	1/7/92 1005		EPP22	~~Field Duplicate~~
MEJK10	2	L	G	3			X					130715	CRMW115-01	1/7/92 1005		EPP22	~~Field Duplicate~~
MEJK11	4	L	G	2	X							130716	CRMWFB-01	1/7/92 1115		EPP23	~~Field Blank~~ B
MEJK11	4	L	G	3			X					130717	CRMWFB-01	1/7/92 1115		EPP23	~~Field Blank~~ B

Shipment for Case complete? (Y/N) N

Page 1 of 1

Sample used for a spike and/or duplicate: MEJK06, MEJK09

Additional Sampler Signatures

Chain of Custody Seal Number: 175458-59

CHAIN OF CUSTODY RECORD

additional bottles would need to be collected if this is the sample to be used for MS and dup.

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Cathy Kouris	1/7/92 1830				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)

EPA Form [illegible]-1 (Rev. 8-91) Replaces EPA Form (2075-6), previous edition which may be used

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6.0 CALIBRATION PROCEDURES AND FREQUENCIES

As an activity which affects data quality, instrument calibration must be done in accordance with formal written procedures. The calibration procedures for CLP RAS analyses are provided in the current EPA CLP SOW OLM01.0 (8/91) for low-medium concentration organics and the current SOW ILM01.0 (9/91) for low-medium concentration inorganics. Calibration procedures for CLP high-concentration organics are provided in that current SOW (9/88) and for CLP high-concentration inorganics are provided in that current SOW IHC01.1. Calibration procedures for SAS analysis are described in the appropriate SAS provided in Appendix B. Calibration procedures for the screening level volatile organic analysis are provided in Appendix E. General requirements for the calibration of instruments are established in the calibration procedures provided in Appendix C. The instrument must be calibrated and maintained by trained personnel to operate within manufacturer's specifications. Field instruments will be calibrated prior to any measurements in the field. Calibration procedures for field instruments are provided in Appendix C. Field instruments will be recalibrated if found to be necessary by performance of QC checks.

Standard Operating Procedures for field and laboratory instruments are described in Section 12.

The Standard Operating Procedures for field instrument calibration to be used during the Southeast Rockford Phase II RI (Appendix C) are detailed in the CDM Site Investigation Procedures Manual (SIPM). These procedures are listed below:

Procedure Title	SIPM Method No.
• Equipment and Instrument Calibration and Maintenance, General	6600001
• Calibration and Maintenance Procedure YSI Model 33 SCT Meter	6617002

Procedure Title	SIPM Method No.
• Calibration and Maintenance Procedures HaakeBuchler pH Stick	6617003
• Calibration Procedure for the HNu PS 101	6607001
• Calibration and Maintenance Procedure Century Systems Portable Organic Vapor Analyzer Model OVA-128	6607003

The calibration for the thermometer will be performed by using an ice/water slurry to check for accuracy. The thermometer shall be within $\pm$ 0.5° of 0°C when the thermometer has equilibrated with the ice/water slurry.

All calibration performed in the field will be documented in the field logbook.

7.0 ANALYTICAL PROCEDURES

For the Southeast Rockford Phase II Remedial Investigation, the analytical procedures for the CLP Laboratory are specified in the current USEPA CLP SOW OLM01.0 (8/91) for RAS low-medium concentration organic analyses, in the current CLP SOW ILM02.0 (9/91) for RAS low-medium concentration inorganic analyses, in the current CLP SOW (9/88) for high-concentration organic analyses and in the current CLP SOW IHC01.1 for high-concentration inorganic analyses. The analytical procedures for SAS CLP Laboratory analysis are specified in the SAS Client Request Forms (Appendix B). The analytical method (SW846 8010) to be used for the screening level volatile organic analysis is provided in Appendix E.

Analytical procedures for field analytical equipment are discussed in the Sampling and Analysis Plan.

Standard analytical procedures for field and laboratory analytical equipment are discussed in Section 12.

8.0 INTERNAL QUALITY CONTROL CHECKS

Internal quality control checks for field instruments are discussed in Section 3.5. Quality control procedures for field measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings and/or by calibrating the instruments (where appropriate). When any field instrument fails the QC checks for calibration it will be re-calibrated, repaired, or replaced, whichever is necessary. Quality control of field sampling will involve collecting field duplicates and blanks in accordance with the applicable procedures described in the SAP.

Internal quality control procedures for RAS from the CLP are specified in the current SOW OLM01.0 (8/91)for organics and the current SOW ILM02.0 (9/91) for inorganics as well as in the method descriptions. Internal quality control procedures for high-concentration organics are specified in that current SOW (9/88) and for high-concentration inorganics are specified in that current SOW IHC01.1. The quality control checks for laboratory instrumentation are discussed in Section 3.3. These specifications include the types of audits required (sample spikes, surrogate spikes, reference samples, controls, blanks), the frequency of each audit, the compounds to be used for sample spikes and surrogate spikes, and the quality control acceptance criteria for these audits.

The levels of internal quality control for SAS from CLP for the drinking water detection level analyses and the total organic carbon, total dissolved solids and total suspended solids analyses are described in the SAS requests in Appendix B of this QAPP.

The level of interval quality control for the screening level volatile organic analysis is provided in Appendix E.

9.0 DATA REDUCTION, VALIDATION AND REPORTING

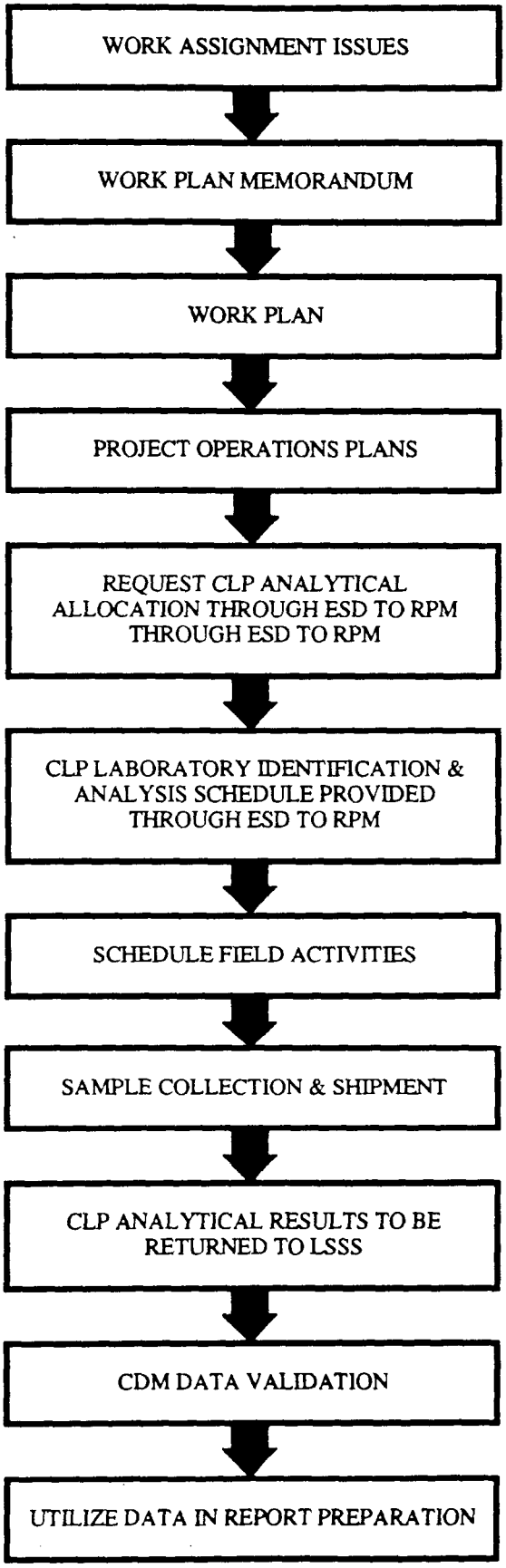
Data reduction, evaluation and reporting of those samples analyzed by CLP laboratories will be performed in accordance with the specifications of the USEPA Contract Laboratory Program. The data management approach for CLP-analyzed samples is illustrated with the logic diagram shown in Figure 9-1. Data reduction for RAS analytical services performed by USEPA CLP will be in accordance with current CLP Statements of Work (SOW) for organics and inorganics. Data reporting for RAS and SAS analytical services will be in accordance with current CLP Statements of Work (SOW) and the SAS requests in Appendix B, respectively.

The requirements for accuracy, precision and completeness for analytical data from USEPA CLP SAS analysis are provided in the SAS client request forms (Appendix B). Data reduction for RAS analytical services at the USEPA CLP will be in accordance with the current CLP Laboratory Statement of Work (SOW). The data will be assessed by verification of the reduction results and confirmation of compliance with QA/QC requirements.

The analytical data from the screening level volatile organic analysis will be evaluated for accuracy precision and completeness in the field. The data will be assessed by reviewing field and laboratory duplicates and blanks and the results will be summarized in the report.

Raw data from field measurements and sample collection activities will be appropriately recorded in the field log book. If the data is to be used in the project reports, it will be reduced or summarized and the method of reduction documented in the report.

FIGURE 9-1 CLP ANALYTICAL DATA MANAGEMENT FLOW CHART



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The CLP RAS and SAS analytical data will be validated by CDM personnel to ensure that the data is sufficient to support the risk assessment and the feasibility study. The data validation procedures that CDM will follow are provided in two USEPA documents. These documents are: National Functional Guidelines for Organic Data Review, 6/91; and Laboratory Data Validation: Functional Guidelines for Evaluating Inorganic Analyses, 7/88. Additionally, specifications provided in the guidelines and/or acceptance criteria provided by the USEPA Central Regional Laboratory QA Section will also be followed.

Twenty-five percent of the analytical data analyzed through the USEPA CLP will be validated by CDM. Samples chosen for validation will be the more critical samples such as background samples and samples from new monitoring wells. The additional data to be validated will be chosen at random from various data sets. If quality problems are encountered within a data set, the remainder of the data set will be validated.

10.0 PERFORMANCE AND SYSTEMS AUDITS

Performance and systems audits may be conducted for activities performed by any entity performing services on this project, including CLP laboratories and field team activities.

Performance and systems audits of field activities may be performed periodically by the CDM QA Manager in accordance with CDM audit procedures, the USEPA Region V Environmental Services Division or the IEPA Project Manager. Audits will be performed to evaluate sampling activities including sample ID, chain-of-custody, field documentation and proper sampling procedures. The results of the field audits will be reported as part of the Quality Assurance Reports to management.

The performance and systems audits of the USEPA CLP Laboratory are the responsibility of USEPA EMSL-LV. For CLP laboratories performing SAS analyses, the audit procedures are the same as those for CLP RAS analyses which are specified in the SOWs for organics and inorganics. Performance audits are used to evaluate laboratory performance. These audits consist of random data audits, continuous trend analyses of laboratory quality control data and quarterly analysis of performance evaluation (PE) samples. Systems audits are performed to verify continuity of personnel, instrumentation and quality control requirements contained in the IFBs. Systems audits are performed by ESML - Las Vegas and consist of annual on-site inspections. In addition to these audits, additional performance audits may be requested in the SAS requests for specific analyses. For laboratories authorized to do SAS analyses only, audit procedures are as specified by the Sample Management Office.

No audits are planned for the screening laboratory (ALS).

11.0 PREVENTIVE MAINTENANCE

All laboratories participating in the CLP are required under respective SOWs for organics and inorganics to have Standard Operating Procedures (SOPs) for preventive maintenance for each measurement system and required support activity. All maintenance activity must be documented in logbooks to provide a history of maintenance records. The preventive maintenance program for the screening laboratory, ALS, is described in Appendix E.

The field equipment to be used for this project includes field pH meters, conductivity meters and thermometers. Preventive maintenance of field analytical equipment used at Southeast Rockford will be conducted in accordance with the maintenance procedures outlined in the Standard Operating Procedures provided in Appendix C. Specific preventive maintenance procedures for this equipment are referenced in Appendix C of the SAP. The Field Manager will be responsible for implementing these procedures, documenting the procedures carried out in the logbook and on the proper forms.

12.0 ANALYTICAL SERVICES

General requirements for analytical procedures are established in the CLP Quality Assurance Programs. These programs establish the need for formally documented procedures which require:

- The use of CLP laboratories and analytical procedures for all enforcement, litigation, and evidentiary data,

or

- The use of the CLP for drinking water analytical procedures for all enforcement, litigation, and evidentiary data, and

- The specification of analytical procedures for all analytical field procedures and non-CLP generated data.

12.1 ROUTINE ANALYTICAL SERVICES LABORATORY PROCEDURES

The current USEPA Contract Laboratory Program Statement of Work (SOW) for organics and the SOW for inorganics specify the analytical procedures to be used for performing the requested organic and inorganic analyses on the soil and water samples collected during the Southeast Rockford Remedial Investigation. Also specified by the SOWs are the sample custody procedures, instrument calibration procedures and frequency of calibration.

12.2 SPECIAL ANALYTICAL SERVICES LABORATORY PROCEDURES

The analytical procedures to be used for performing the Special Analytical Services (SAS) analyses are described in the SAS requests in Appendix C of this QAPP. Also specified in

the SAS requests are calibration procedures, frequency of calibration and the internal quality control checks required for each analysis. The SAS specifications also include the types of audits required (sample spikes, surrogate spikes, reference samples, control blanks), the frequency of each audit, the compounds to be used for sample spikes and surrogate spikes, and the quality control acceptance criteria for these audits.

12.3 SCREENING LEVEL SERVICES LABORATORY PROCEDURES

The analytical procedure to be used for performing the screening level volatile organic analysis is SW846 8010 and is contained in Appendix E of the QAPP.

12.4 FIELD SCREENING ANALYTICAL PROCEDURES

For field screening analyses, the relevant SOPs are found in the CDM Site Investigation Procedures Manual as follows:

Procedure Title	SIPM Method No.
• Operation Procedure YSI Model 33 SCT Meter	5617002
• Operation Procedure for HaakeBuchler pH Stick	5617003
• Procedure for Determining Temperature of Groundwater	5617004
• Operation procedure for HNu Model PS 101 photoionization analyzer	5607001
• Operation procedure for Century portable organic vapor analyzer model OVA-128	5607003
• Procedure for Filtration of Samples	5617007

All procedures used and results obtained will be documented in the field logbook.

13.0 CORRECTIVE ACTION

Corrective action for the CLP is implemented at several different levels. The laboratories participating in the CLP are required to have a written SOP specifying corrective action to be taken when an analytical error is discovered or the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification to the analyst of the error and correct procedures.

Corrective action for SAS requests will be implemented as required by the specific SAS request as well as standard CLP procedures.

During field operations including the screening level VOC analysis, if any nonconformance with established quality control procedures is identified, the Project Manager will be responsible for developing and initiating corrective action. The IEPA Project Manager will be responsible for reporting any proposed, developed or initiated corrective actions to the USEPA Region V Project Officer for review and approval. Corrective action needed for on-site activities will be initiated by the field team leader, but must be approved by the Project Manager.

14.0 QUALITY ASSURANCE REPORTS TO THE MANAGEMENT

The QA reports will be a part of the regular quarterly project reports that the IEPA submits to the USEPA Region V RPMS. The QA reports will contain (but not be limited to) project status, results of performance and systems audits, data quality assessments, quality assurance problems with proposed corrective actions and QAPP amendments.

15.0 GLOSSARY OF TERMS

ACCURACY - The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted referenced or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100 (X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in a system.

AUDIT - A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) system audits that consist of a review of the quality control system to ensure that a comprehensive set of quality control methods, procedures, reviews, and signoff approvals is established or in place, and (2) performance audits in which project activities are observed in process for their compliance with the established quality control procedures and requirements.

COMPARABILITY - Expresses the qualitative confidence with which one data set can be compared to another. For this project, the data comparability will be achieved by the following:

a. Analytical results will be reported in appropriate units;

b. Same or similar sampling procedures used in previous investigations will be used; and

c. Quality assurance and quality control requirements will be observed.

COMPLETENESS - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions to characterize the environmental condition and/or the amount of valid data obtained from the measurements system compared with the amount of data that was expected under normal conditions. Percent completeness is defined as:

$$\frac{\text{(number of valid data)}}{\text{(number of samples collected for each parameter analyzed)}} \times 100$$

DATA VALIDATION - A systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, certification, and review.

PRECISION - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".

QUALITY ASSURANCE - The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement effort to meet user requirements.

QUALITY ASSURANCE PROGRAM PLAN - An orderly assemblage of management policies, objectives, principles, and general procedures by which an agency or laboratory outlines how it intends to produce data of known and accepted quality.

QUALITY ASSURANCE PROJECT PLAN - An orderly assemblage of detailed and specific procedures which delineates how data of known and accepted quality are produced for a specific project. (A given agency or laboratory would have only one quality assurance

program plan, but would have a quality assurance project plan for each of its projects.)

QUALITY CONTROL - The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

REPRESENTATIVENESS - A qualitative measure of the degree to which the data accurately and precisely represents a characteristic of a population, parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative criteria which is associated with the proper design of a sampling and analysis program.

STANDARD OPERATING PROCEDURE - A written document which details an operation, analysis, or action whose mechanisms are thoroughly prescribed and which is commonly accepted as the method for performing certain routine or repetitive tasks.

APPENDIX A
EXISTING DATA SUMMARY

EXISTING DATA SUMMARY

Existing data for the Southeast Rockford site is summarized in the tables on the following pages. The locations of the samples are shown on Figure 4-9 and 4-10 of the Technical Memorandum for Phase I Field Activities completed for IEPA by CDM in October 1992 (or Drawing 1 of the Phase II Work Plan). Table 4-2 gives results for volatile organics, while Table 4-3 summarizes the results for semivolatile organics and pesticides as well as volatiles. Table 4-4 summarizes the results for inorganic constituents. The sampling results are discussed in greater detail in Section 4 of the Technical Memorandum.

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-4-91	9-4-91	9-4-91	9-4-91	9-4-91	9-5-91
Sample Number	MW101B	MW101A	MW102A	MW102B	MW102C	MW103A
Organic Traffic Report Number	EPS-01	EPS-03	EPS-06	EPS-07	EPS-09	EMF-17
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone						
1,1-Dichloroethene	940 E	65	1 J		32	68 D
1,1-Dichloroethane	2900 E	170	11 J		89	200 D
cis-1,2-Dichloroethene	2800 E	250	7 J		68	690 D
trans-1,2-Dichloroethene	55					6
Chloroform	130		0.9 J			11
1,2-Dichloroethane	62					
2-Butanone						
1,1,1-Trichloroethane	12000 E	710	19 J		92	670 D
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene	3600 E	180	1 J		26	210 D
Dibromochloromethane						
1,1,2-Trichloroethane	60					4
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene	1200 E				4 J	110 E
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank E=The reported result exceeded the calibration range of the analytical run D=The result reported is from a re run of the sample at a higher dilution R The data is unuseable

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Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-5-91	9-5-91	9-5-91	9-6-91	9-6-91	9-6-91
Sample Number	MW103B	MW103C	MW104A	MW104C	MW104B	MW105B
Organic Traffic Report Number	EMF-18	EMF-21	EMF-25	EMF-23	EMF-28	EMF-30
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						5 J
Chloroethane						
Methylene Chloride						
Acetone						
1,1-Dichloroethene	190	140		1	2	4 J
1,1-Dichloroethane	140	140		1	4	78
cis-1,2-Dichloroethene	130	170				110
trans-1,2-Dichloroethene						4 J
Chloroform	24 J	22 J				
1,2-Dichloroethane						13
2-Butanone						
1,1,1-Trichloroethane	1000	1200	0.7 J	9 J	12	81
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene	380	380		0.6 J		25
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene	130	110				
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						
	1994	2162	0.7	11.6	18	320

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re-run of the sample at a higher dilution. R=The data is unusable.

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-6-91		9-6-91	9-9-91	9-9-91	9-9-91
Sample Number	MW105A	MW105C	MW105D	MW106A	MW106B	MW107A
Organic Traffic Report Number	EMF-32	EMF-35	EMF-40	EMF-43	EMF-46	EMF-47
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone						
1,1-Dichloroethene				94		
1,1-Dichloroethane	25	6	5	250	3	
cis-1,2-Dichloroethene	55	5	5	4100	4	
trans-1,2-Dichloroethene	2 J					
Chloroform		5				
1,2-Dichloroethane		1	1			
2-Butanone						
1,1,1-Trichloroethane	24	3 J		6000	6 J	110
Bromodichloromethane		1				
1,2-Dichloropropane						
Trichloroethene	9	0.9 J			8	
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene	2 J			43 J	3	
1,1,2,2-Tetrachloroethane						
Toluene				59 J		
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						
	11 7	21.9	11	19546	24	

Data Qualifiers: J-The value presented is an estimated value. B-The analyte is also found in the associated lab blank. E-The reported result exceeded the calibration range of the analytical run. D-The result reported is from a re run of the sample at a higher dilution. R-The data is unuseable

US4322

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-9-91	9-9-91	9-9-91	9-10-91	9-10-91	9-10-91
Sample Number	MW107B	MW107C	MW106C	MW109C	MW109B	MW109A
Organic Traffic Report Number	EMF-49	EPS-11	EPS-16	EPS-18	EPS-24	EPS-26
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride		2 B				
Acetone						
1,1-Dichloroethene	1					
1,1-Dichloroethane	1		3			
cis-1,2-Dichloroethene			2			
trans-1,2-Dichloroethene						
Chloroform						
1,2-Dichloroethane			0.9 J			
2-Butanone						
1,1,1-Trichloroethane	36		2 J		2 J	0.6 J
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene			2			
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene			1			
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

10.9

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re-run of the sample at a higher dilution. R=The data is unuseable.

US4323

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-10-91	9-10-91	9-10-91	9-10-91	9-11-91	9-11-91
Sample Number	MW108B	MW110A	MW108C	MW108A	MW110B	MW110C
Organic Traffic Report Number	EPS-27	EPS-30	EPS-32	EPS-34	EPS-36	EPS-41
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone		R		R		R
1,1-Dichloroethene						
1,1-Dichloroethane	0.6 J		0.6 J			
cis-1,2-Dichloroethene						
trans-1,2-Dichloroethene						
Chloroform					2	
1,2-Dichloroethane						
2-Butanone		R		R		R
1,1,1-Trichloroethane	1 J			3 J	2	
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene				0.5 J		
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene						
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J-The value presented is an estimated value. B-The analyte is also found in the associated lab blank. E-The reported result exceeded the calibration range of the analytical run. D-The result reported is from a re run of the sample at a higher dilution. R - The data is unuseable

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-11-91	9-11-91	9-11-91	9-12-91	9-12-91	9-12-91
Sample Number	MW111A	MW111B	MW111C	MW11	MW27	MW28
Organic Traffic Report Number	EPS-45	EPS-47	EPS-49	EPS-53	EPS-55	EPS-58
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone						
1,1-Dichloroethene	4	9		7		50
1,1-Dichloroethane		1		20		100
cis-1,2-Dichloroethene	15	40		23		100
trans-1,2-Dichloroethene						
Chloroform				1		
1,2-Dichloroethane				0.7	J	
2-Butanone						
1,1,1-Trichloroethane	19	48		41	J	170
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene	11	14		18		67
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene	12	15		0.6	J	
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re run of the sample at a higher dilution. R=The data is unuseable

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-12-91	9-12-91	9-13-91	9-13-91	9-16-91	9-16-91
Sample Number	MW23	MW26	MW24	MW14	MW20	MW22
Organic Traffic Report Number	EPS-61	EPS-63	EPS-65	EPS-67	EPM-31	EPS-72
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane					5	
Methylene Chloride						
Acetone						
1,1-Dichloroethene					9	0.8 J
1,1-Dichloroethane	4				140 D	3
cis-1,2-Dichloroethene	3	3			35 D	3
trans-1,2-Dichloroethene						
Chloroform						
1,2-Dichloroethane						
2-Butanone						
1,1,1-Trichloroethane	16		2		36 D	22
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene	2				4	2
Dibromochloromethane		1				
1,1,2-Trichloroethane					1	
Benzene					5	
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene						
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re run of the sample at a higher dilution. R=The data is unuseable

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-16-91	9-16-91	9-16-91	9-16-91	9-17-91	9-17-91
Sample Number	MW16	MW30	MW17	MW21	DECON	MW9
Organic Traffic Report Number	EPS-74	EPS-76	EPS-78	EPS-83	EPN-51	EPS-87

Volatile Organics (ug/L)

Chloromethane												
Bromomethane												
Vinyl Chloride												
Chloroethane												
Methylene Chloride												
Acetone												
1,1-Dichloroethene	30	D	0.6	J	4		1					
1,1-Dichloroethane	52	D	14		16		14				0.9	J
cis-1,2-Dichloroethene	64	D	11		8		15				2	
trans-1,2-Dichloroethene	1		2				1					
Chloroform									3	J	0.7	J
1,2-Dichloroethane	2		0.5	J			0.6	J				
2-Butanone												
1,1,1-Trichloroethane	180	BD	50	D	15	J	31	D				
Bromodichloromethane									2			
1,2-Dichloropropane					0.5	J						
Trichloroethene	48	D	17		2		13				2	J
Dibromochloromethane									0.9	J		
1,1,2-Trichloroethane	1											
Benzene												
Trans-1,3-Dichloropropene												
Bromoform												
4-Methyl-2-Pentanone												
2-Hexanone												
Tetrachloroethene	2											
1,1,2,2-Tetrachloroethane												
Toluene												
Chlorobenzene												
Styrene												
1,2-Dibromo-3-Chloropropane												
1,3-Dichlorobenzene												
1,4-Dichlorobenzene												
1,2-Dichlorobenzene												

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re-run of the sample at a higher dilution. R=The data is unuseable

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-17-91	9-17-91	9-17-91	9-17-91	9-30-91	9-30-91
Sample Number	MW12	MW29	MW31	MW32	MWS32A	MWS23
Organic Traffic Report Number	EPS-89	EPS-91	EPS-93	EPS-97	EPM-36	EPM-38

Volatile Organics (ug/L)

Chloromethane												
Bromomethane												
Vinyl Chloride												
Chloroethane												
Methylene Chloride												
Acetone												
1,1-Dichloroethene					16		2		81		150	
1,1-Dichloroethane		1			42	D	9				110	
cis-1,2-Dichloroethene		2			49	D	7				20	J
trans-1,2-Dichloroethene					0.6	J						
Chloroform		0.8	J		4	J						
1,2-Dichloroethane					1							
2-Butanone												
1,1,1-Trichloroethane					120	D	14	J	340		770	
Bromodichloromethane												
1,2-Dichloropropane												
Trichloroethene		2	J		30	D	2		36		53	
Dibromochloromethane												
1,1,2-Trichloroethane												
Benzene												
Trans-1,3-Dichloropropene												
Bromoform												
4-Methyl-2-Pentanone												
2-Hexanone												
Tetrachloroethene					0.8	J			50		160	
1,1,2,2-Tetrachloroethane												
Toluene												
Chlorobenzene												
Styrene												
1,2-Dibromo-3-Chloropropane												
1,3-Dichlorobenzene												
1,4-Dichlorobenzene												
1,2-Dichlorobenzene												

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re run of the sample at a higher dilution. R=The data is unuseable

US4328

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-30 91	9-30-91	10-1-91	9-30 91	10-1-91	10-1-91
Sample Number	MWS29	MWS33	MWS33A	MWS34A	MWS28A	MWS34
Organic Traffic Report Number	EPM-41	EPM-45	EPM-47	EPM-49	EPM-52	EPM-54
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone						
1,1-Dichloroethene						
1,1-Dichloroethane	0.6 J					2
cis-1,2-Dichloroethene						
trans-1,2-Dichloroethene						
Chloroform						
1,2-Dichloroethane						
2-Butanone						
1,1,1-Trichloroethane	0.9 J	2		2	11	19
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene						2
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene		0.6 J				
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene						
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re run of the sample at a higher dilution. R=The data is unuseable

US4329

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	10-1-91	10-1-91	10-1-91	10-1-91	10-2-91	10-1-91
Sample Number	MWS9	MWS41	MWS37	MWS10	MWS31A	MWS40
Organic Traffic Report Number	EPM-56	EPM-58	EPM-60	EPM-62	EPM-67	EPM-69
Volatile Organics (ug/L)						
Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone						
1,1-Dichloroethene	43			4	3	
1,1-Dichloroethane					0.6 J	
cis-1,2-Dichloroethene						
trans-1,2-Dichloroethene						
Chloroform					1	
1,2-Dichloroethane						
2-Butanone						
1,1,1-Trichloroethane	400			18	84 J	
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene	22 J			1		
Dibromochloromethane						
1,1,2-Trichloroethane						
Benzene						
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene	25			2	0.6 J	
1,1,2,2-Tetrachloroethane						
Toluene						
Chlorobenzene					12	
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re run of the sample at a higher dilution. R. The data is unuseable

US4330

Table 4-2. Volatile Organic Results for Groundwater Samples, Southeast Rockford

Date Sampled	9-13-91	10-2-91	10-2-91
Sample Number	MW15	MWS5	MWS32
Organic Traffic Report Number	EPS69	EPM74	EPM76

Volatile Organics (ug/L)

Chloromethane						
Bromomethane						
Vinyl Chloride						
Chloroethane						
Methylene Chloride						
Acetone		R				
1,1-Dichloroethene	4		340		240	E
1,1-Dichloroethane	16		2100	E	4	
cis-1,2-Dichloroethene	13		250		4	
trans-1,2-Dichloroethene					2	
Chloroform	0.6	J			0.5	J
1,2-Dichloroethane					2	
2-Butanone						
1,1,1-Trichloroethane	32	E	1300	E	470	E
Bromodichloromethane						
1,2-Dichloropropane						
Trichloroethene	5		50		240	E
Dibromochloromethane						
1,1,2-Trichloroethane					4	
Benzene			32	J		
Trans-1,3-Dichloropropene						
Bromoform						
4-Methyl-2-Pentanone						
2-Hexanone						
Tetrachloroethene			120		110	E
1,1,2,2-Tetrachloroethane						
Toluene			14000	E	6	
Chlorobenzene						
Styrene						
1,2-Dibromo-3-Chloropropane						
1,3-Dichlorobenzene						
1,4-Dichlorobenzene						
1,2-Dichlorobenzene						

Data Qualifiers: J=The value presented is an estimated value. B=The analyte is also found in the associated lab blank. E=The reported result exceeded the calibration range of the analytical run. D=The result reported is from a re run of the sample at a higher dilution. R The data is unuseable

US4331

Table 4-3
A Comparison of the Organic Groundwater Data at Southeast Rockford to Federal and State MCLs (all results in ppb)

Compound (a)	Federal MCL	Illinois MCL	Range of Phase I samples	Proportion of Phase I samples with detectable concentrations (b)	Proportion of Phase I samples exceeding MCLs
Volatile Organics					
Vinyl Chloride	2	2	<1-5J	1/68 (1.5%)	1/68 (1.5%)
Chloroethane			<1-5	1/68 (1.5%)	
1,1-Dichloroethene	7	7	<1-940E	30/68 (44.1%)	19/68 (27.9%)
1,1-Dichloroethane			<1-2,900E	35/68 (51.5%)	
cis-1,2-Dichloroethene	70		<1-4,100	35/68 (51.5%)	9/68 (13.2%)
trans-1,2-Dichloroethene	100		<1-55	8/68 (11.8%)	none
Chloroform	100 (c)		<1-130	9/68 (13.2%)	1/68 (1.5%)
1,2-Dichloroethane	5	5	<1-62	7/68 (10.3%)	2/68 (2.9%)
1,1,1-Trichloroethane	200	200	<1-12,000E	47/68 (69.1%)	11/68 (16.2%)
Trichloroethene	5	5	<1-3,600E	34/68 (50%)	24/68 (35.3%)
1,1,2-Trichloroethane			<1-60	5/68 (7.3%)	
Benzene	5	5	<1-32J	2/68 (2.9%)	2/68 (2.9%)
Tetrachloroethene	5		<1-1,200E	18/68 (26.5%)	13/68 (19.1%)
Toluene	1000		<1-14,000E	3/68 (4.4%)	1/68 (1.5%)
Chlorobenzene	100	100	<1-12	1/68 (1.5%)	none
Semi-volatile Organics					
2-Methylphenol			<10-85	1/65 (1.5%)	
4-Methylphenol			<10-54	2/65 (3.1%)	
2,4-Dimethylphenol			<10-12	1/65 (1.5%)	
di-n-Butylphthalate			<10-10	1/65 (1.5%)	
Pesticides					
gamma BHC (Lindane)	0.2	4	<0.01-0.082J	1/62 (1.6%)	none
Heptachlor	0.4	0.1	<0.01-0.039J	1/62 (1.6%)	none
Heptachlor epoxide	0.2	0.1	<0.01-0.079J	1/62 (1.6%)	none
Endrin	0.2	0.2	<0.02-0.19J	1/62 (1.6%)	none

(a): Results are given for any compound detected at greater than or equal to 25% of the more conservative MCL, or any compound reported at greater than or equal to the Contract Required Quantitation Limits (CRQL)

(b) Detectable concentrations are those which are equal to or exceed the CRQL

(c) Denotes standard for total trihalomethanes, which comprise chloroform, bromoform, dibromochloromethane, and bromodichloromethane

Table 4-4
Comparison of Inorganic Groundwater Results at Southeast Rockford to Federal MCLs and Illinois Groundwater Quality Standards (all values in ppb)

Compound (b)	Federal MCL	Illinois Standards*	Range of Phase I samples	Proportion of Phase I samples with detectable concentrations (c)	Proportion of Phase I samples exceeding MCLs
Aluminum	50-200**	-	<32 - 3,340	41/51 (80.4%)	22/51 (43.1%)***
Iron	300**	5,000	<6 - 5,220	42/51 (82.3%)	11/51 (21.6%)
Manganese	50**	150	<2 - 667	44/51 (86.3%)	19/51 (37.2%)
Arsenic	50	50	<1 - 44.4	13/51 (25.5%)	none
Selenium	10	50	<1 - 6.5	4/51 (7.8%)	none

(a):Results are given for filtered groundwater samples only.

(b): Results are given for any compound detected at greater than or equal to 50% of the more conservative MCL.

(c): Detectable concentrations are those which are equal to or exceed the Instrument Detection Limit.

* : Illinois Groundwater Quality Standards, 35 Ill. Adm. Code, Subpart C, Section 620.410, November 7, 1991.

**: Indicates secondary MCL (SMCL), recommended but not promulgated.

***: Indicates the number of exceedences of the more conservative MCL.

APPENDIX B

SAS REQUEST FORMS

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virgina 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: V/CDM

B. RSCC Representative: Jan Pels

C. Telephone Number: 353-2720

D. Date of Request: March 1993

E. Site Name: S.E. Rockford Groundwater

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of ambient air samples for volatile organics using EPA TO-14. Laboratory to provide prepared Summa canisters.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

34 samples - 28 investigative, 3 field blanks and 3 field duplicates for low concentration VOCs in air.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund Remedial

4. Estimated date(s) of collections: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples:

   30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program: EPA TO-14. If the laboratory proposes to use GC-selective detectors (ECD, FID, PID, Hall), a confirmational analysis is required for detected compounds. This may be accomplished using GC/MS-Full Scan, GC/MS in the selected ion monitoring mode using ions listed in Table 2 of TO-14, using another selective detector with the necessary sensitivity, or by second column confirmation. Ten percent of the samples per SDG containing detectable analytes will require confirmation. All samples containing detectable quantities of vinyl chloride will require confirmation for this compound only. If GC/MS-Full Scan is used for confirmation up to 10 non-target peaks should be tentatively identified.

   Also see Attachment A

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

   The laboratory performing analysis will supply and prepare the Summa canisters. See Attachment 1.

9. Analytical results required (if known, specify format for data sheets, QA/OC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

   See Attachment 2.

   Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

    See Attachment C.

11. Name of sampling/shipping contact: Yan Yan Ma

    Phone: 312-474-1313

10504.13

US4336

I. DATA REQUIREMENTS

Parameter	Detection Limit (ppbv)	Precision Desired (+% or Conc.)
Vinyl Chloride	0.2	± 20%
1,1,1-Trichloroethane	0.1	
Trichloroethylene	0.1	
Tetrachloroethylene	0.1	
1,2-Dichloroethylene	0.5	
1,1 Dichloroethane	0.5	
1,2 Dichloroethane	0.5	
1,1 Dichloroethylene	0.5	

II. QC REQUIREMENTS

Adults Required	Frequency of Audits	Limits* (% or Conc.)
See Attachment 1		
As per EPA TO-14		

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

Attachment 1

Canister Cleaning and Verification: Canister cleaning and verification is a requirement. After cleaning, it is considered acceptable if no individual analyte is present at a detectable concentration. After cleaning, ten percent (10%) of the canisters will be analyzed for verification of the cleaning procedure. The canister selected for verification will be the one from the sample delivery batch that had previously contained the highest overall concentration of analytes. If no analytes are detected, the entire batch is suitable for shipment. Otherwise, the canisters must either be individually verified, cleaned as necessary and then verified again by the previously mentioned criteria or by recleaning the entire batch and then the canister that had previously failed will be verified following the previously mentioned criteria.

The canister cleaning deliverables are as follows:

- Form I results.
- Raw data/chromatograms.
- Low standard at 0.5 ppbv for the compounds of interest.
- System blank showing those compounds to be less than 0.2 ppbv.

Canister cleaning guidelines are described below:

1. EPA Method TO-14 outlines the most desirable method of cleaning.

2. Any method used must be capable of evacuating the canister to less than 0.05 mm Hg (50M Torr).

3. Any method not utilizing an evacuation/pressure cycle must include heating of the canister to no greater than 160°C but no less than 100°C during evacuation.

4. Any method not utilizing an evacuation/pressure cycle must maintain an evacuation of less than 0.05 mm Hg (50m Torr) for no less than 12 hours.

5. All vacuum pumps utilized in this procedure must include a suitable trap for the prevention of backstreaming.

6. All connections to canisters must be made with brass compression fittings to avoid damage to the canister inlet fitting.

QA/QC Requirements: Holding Time < 30 days from sample collection.

Initial Calibration: A five (5) point calibration at 0.5, 1.0. 3.0, 5.0, and 10 ppbv. Percent coefficient of variation (%CV) or percent relative standard deviation (%RSD) of the response factors (RF) must be less than 5.0%.

Retention Time Windows: See Analytical Method TO-14.

Continuing Calibration: Daily continuing calibration at 1.0 ppb in triplicate. If the response value of the 1.0 ppb calibration analysis differs significantly from the initial calibration response factor (RF) mean at the -.05 level of significance of a 2-tailed t test, a new initial calibration is to be analyzed.

After any change to the system that may alter the signal response which includes but is not limited to: column or carrier gas change, bakeout, or detector cleaning, a daily continuing calibration must be run. If this calibration fails to meet the previously mentioned criteria for daily calibration, a new initial calibration must be run.

Surrogate Standards: Fifty microliters of approximately 5 ppm in zero grade air of Bromochloromethane and 1, 2-Bromochloropropane will be spiked into all blanks, standards, samples, and sample duplicates. Other surrogates may also be used. The laboratory must ensure that surrogates do not co-elute with analytes.

System Blank: A system blank will be run after any calibration. For blank analysis use humidified Zero Grade air from a known and consistent source. Identify source on Form 4. The blank must be free of interfering peaks and of volatile organics greater than 0.2 ppbv. To prevent carryover, a system blank must be run after any analysis in which any analyte saturates the detector.

Duplicates: In each batch, one sample for duplicate analysis will be designated. The maximum allowable relative percent difference for each analyte whose concentrations are 3 times greater than the LOD will be 10.0

percent. The maximum allowable relative percent difference for each analyte whose concentrations are less than 3 times the LOD will be 20.0 percent. Failure to meet this criteria will require a reanalysis of the sample.

Identification Criteria: Analytes will be qualitated by matching the retention time windows of the sample and the standard. In the unlikely event of overlapping retention time windows, the laboratory will use the peak whose retention time most closely matches that of the sample. Confirmational analysis will be required for all identified peaks with overlapping RT windows.

Standard Operating Procedures (SOP): All methods and procedures related to the analysis and cleaning of SUMMA passivated canisters should be documented in a logical, step-wise manner. These should include but not be limited to: assembly, calibration, maintenance, and operation of specific systems and equipment, preparation, storage, shipment and handling of samples; canisters cleaning and verification; and all aspects of data recording and processing including hardware and software documentation. Canister sample pressure will be immediately recorded after sampling and immediately before analysis. Chain of custody seals will be placed on each sample shipment.

Gas Chromatography/Mass Spectrometry - Full Scan:

All QA/QC requirements for gas chromatography are applicable to GC/MS-full scan analysis (except GC sections on confirmational analysis, detection limits, and identification criteria). In addition, the following QA/QC requirements are applicable to GC/MS analysis:

Instrument Tuning: Each day, prior to initiating any analysis, the gas chromatograph/mass spectrometer (GC/MS) will be tuned with 50 ng. direct column injection of p-Bromofluorobenzene (BFB) as outlined in this section for TO-14. The main exception to the indicated procedures is the frequency of tuning, which will be performed once in 12 hours or the total period of analysis, whichever is more frequent. Tuning information will be provided on Form 5.

Detection Limits: Instrument detection limits are established by demonstrating the ability of the instrument to detect and produce a complete enhanced or unenhanced mass spectrum in which fragmentation ions greater than 20 percent of base peak must be present from a 500 ml sampling of 5 ppbv standard (approximately 2.0 μg depending on the analyte). Determination of detection limits should follow the guidelines in CFR 40, Part 136, Appendix B.

Identification Criteria: Criteria for identification of the compounds found in Attachment #3 is based upon retention time (RT) matching and mass spectrum matching with the standard. The relative retention time (RRT) must be within $\pm$0.06 RRT of the calibration standard, where RRT is the RT of the hit compound in relation to the RT of the corresponding internal standard. Mass spectra must match according to the following criteria:

1. All m/e greater than 15 percent in the standard spectrum must be present in the sample spectrum.

2. The relative intensities of ions must agree within 20 percent between standard and sample spectra.

3. Fragmentation ion greater than 15 percent in the sample spectrum but not in the standard spectra must be accounted for (background, co-eluting peak, etc.).

The laboratory will also perform NIST (NBS) library searches on all peaks that are greater than 10 percent in height than the previous closest internal standard. The searches will be based on relative fit. The analyst will use the criteria established for spectral matching for TCL compounds to identify TIC (Tentatively Identified Compounds), with the exception that the comparison will be made with the NIST Library searches. A certain amount of flexibility is allowed for professional judgment and interpretation of spectra by the analyst. Those peaks which cannot be identified by the above criteria are reported as unknown. The quantitation is expressed as the percent of peak height of target compound in reference to the peak height of the closest previous internal standard.

Attachment 2

Deliverables:

Narrative:

Date samples arrived at the lab.
Sample numbers.
Sampling/analytical problems.
Discussion of any QA/QC outside of the acceptance criteria.

Results:

Report on a Form I equivalent in ppbv analyte/sample.
Volume of sample analyzed.
Include all raw data, chromatograms/mass spectra and quantitation.
Reports for each sample.

Calibration:

Initial calibration with 5 points: 0.5, 1.0, 3.0, 5.0, and 10 ppbv.
Report the calculated: RF, mean retention time, standard deviation, % coefficient of variation and the RT window (±3%).
Daily continuing calibration.
All raw data for the initial and continuing calibrations.
Calculate %D.
Chromatograms and area results/quantitation report.
Date and time of analysis.
Report on CLP forms 6 and 7 equivalent.

Blanks:

Method - daily.
Date and time of analysis.
Report on CLP Form 4 equivalent.
Include all raw data.

Duplicate:

Each batch will contain one sample for duplicate analysis.
Date and time of analysis.
Calculate and report the RPD.
Report on CLP Form 3 equivalent.

Surrogates:

Spike into all blanks, standards, samples, and sample duplicates.
Surrogate retention times.
Report the area and the % recovery.
Report on CLP Form 2 equivalent.

Documentation:

Copy of chain of custody.
Copy of airbill.

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V
B. RSCC Representative: Jan Pels
C. Telephone Number: (312) 353-2720
D. Date of Request: March 1993
E. Site Name: Southeast Rockford Phase II Remedial Investigation

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of soils/sediments/solids by Toxicity Characteristic Leaching Procedure (TCLP) -- Method 1311 of Federal Register June 29, 1990 for regulated semi-volatile TC Constituents and certain deferred semi-volatile TC constituents. Analysis of TC extracts will be done by organic SOW 2/88 modified extraction and final analysis of TC extract extracts by SOW 2/88 or the current revision of SOW OLM. Use a minimum sample aliquot to determine compliance with TCLP regulatory levels. TCLP sample extraction must be done within 14 days of date of sample collection. Two discrete extracts will be generated and analyzed separately for each sample; results from each of the two extractions will be combined on one Form I for each sample.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

5 total investigative soil/sediment/solid samples (includes field duplicates)

3. Purposes of analysis (specify whether Superfund [Remedial or Enforcement], RCRA, NPDES, etc.):

Superfund Remedial

US4344

4. **Estimated date(s) of collection:**

April 1993

5. **Estimated date(s) and method of shipment:**

Daily by overnight carrier

6. **Number of days analysis and data required after laboratory receipt of samples:**

30

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

TCLP Extraction per Method 1311 and analysis of extracts per the current revision of the organic SOW OLM with the modifications listed below and in other sections/attachments of this SAS:
Initial Base/Neutral Extraction at >pH12 will be done using 1,000 mls TCLP extract. 50mls of the water, diluted to 1,000 mls, will be used for acid extraction at pH 1.9-2.0. to minimize poor performance noted in past for acid compounds. Analysis of each extract will be done for complete Table I constituent list. If constituents are found in both extracts, their concentrations will be summed for Form I.
See Attachment I and Table 1.

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

8. **Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

Soils will be collected as 1 quart Bottle for organics. Case narrative should discuss any sample preparation problems. If interstitial water is present when the sample arrives at the laboratory, please premix water with soil prior to initiation of Method 1311. Extra 1-quart Bottles will be provided for preparation of the MS/MSD.

CASE NARRATIVE MUST DISCUSS ANY SAMPLE PROBLEMS

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody documentation, etc.). If not completed, format of results will be left to program discretion.**

All procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain of custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

10. **Other (use additional sheets or attach supplementary information, as needed):**

Attachment I and Method 1311 provide information required for modified extraction of TCLP extract. Use SOW 2/88 or OLM01 for providing Table 1 constituents. Separate Form I's are to be provided for Table 1 constituents and for Table I constituents corrected for MS/MSD recoveries from TCLP extracts. Form I's will also include extraction information of Items "a" through "e" and Item "g" of Attachment I.
Lab must submit all original field documentation (COCs, tags, SAS PLs, etc.) and originals of data to the Region in the time frame referenced in Section 6 of this SAS (analogues to a RAS-CSF).

11. **Name of sampling/shipping contact:** Yan Yan Ma
Phone: (312) 474-1313

I. DATA REQUIREMENTS

Parameter	Detection Limit	Duplicate Precision Desired (+% or Conc.)
see table 1 surrogates of sow 2/88 or olm01 are required at 2 different concentraction levels.	See table 1	see sow 2/88 or Olm01 for ms/msd criteria for extracts.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required TCLP Extraction	Frequency of Audits	Limits (% or Conc.)
Prep. blank for Extract Fluid #1 (see 7.1.4.4 of Method 1311)	Each set of solid samples prepared.	<%5 of Regulatory Levels of Table 1. Discuss in case narrative if larger than limits of appropriate SOW.
Prep. Blank for Extract Fluid #2, if necessary.	Each set of solid samples prepared.	same as above.
Analysis of TCLP Extracts:		
Method or prep. blank for TCLP Extract Determinations.	Per approp. SOW and set up with each TCLP extract batch.	Per approp. SOW for Table 1
Surrogates for SOW 2/88 (at 2 different concentrations levels - 100 ug/l for base neutrals and 2,000 ug/l for acids).	Each sample extract and blank extract.	Per SOW 2/88 or OLM01.
MS/MSD (see table 1)	see Table 1 (One for every ten or fewer samples)	Advisory - used to correct for MS recovery. See note 1 of Table 1.
All other QC audits per SOW 2/88 or OLM01, with surrogates and MS/MSD concentrations at 2 different levels (see Attachment 1 and Table 1).	per SOW 2/88 or OLM01. MS/MSD's will be done at frequency of 1 per 10 sample extracts	per SOW 2/88 or OLM01.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED: FOR CORRECTIVE ACTION AND REANALYSIS, CONTACT THE SAMPLE MANAGEMENT OFFICE.

Reanalysis of TCLP extracts may be necessary per requirements of Note 1 to Table 1. Contact with SMO is mandatory.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT I

Semi-Volatile TCLP Extraction will be done by Federal Register of June 29, 1990 bottle extraction semi-volatile constituents. Samples will be soils, wet soils, sediments, or wet or moist solids, liquids, etc; therefore, the filtration procedure (Section 7.1.1.7 of attached procedure) may produce interstitial water for sediments/soils. Also, any water collecting on top of sediment or soil is not to be discarded, but mixed with sample prior to filtration or % solid determination (Section 7.1.1). TCLP extracts may be a combination of liquid filtrate and solid TCLP extracts (see Sections 7.2.13.2 and 7.3.14) but will depend on the physical nature of soils and sediments collected. Also see Section 8 of SAS request. Particle size reduction is not expected to be necessary for soils and sediments. Sample preparation logs will be needed to record all required information of Method 1311 including (but not limited to):

a. Weight(s) of extracted samples (100g minimum aliquot size for 100% solids content) and volume of any filtrate (Sections 7.2.2 and 7.2.5).
b. Preliminary evaluations of percent (%) solids.
c. pH data for selection of Extraction Fluid #1 or #2 (Section 7.1.4.2)
d. Dates of each preparation step, with associated weights and measured volumes.
e. pH value of final TCLP extract (Section 7.2.14)
f. Holding times (Section 7.4) are to be met and are to be counted from the data of collection using overnight carrier; and
g. Record HCL normality (Section 5.3) and acetic acid normality (Section 5.7.2) (SAS par. 8) and measured pH of Extraction fluids (Section 5.7.1 and 5.7.2). Record dates of each of 4 required measurements.
h. **Standardization of hydrochloric acid and acetic acid for Extraction Fluid #1 and Extraction Fluid #2:**
   1. The 1 N HC1 can be and will be standardized to 1.0 N HC1 $\pm$ 5%.
   2. The pH of Extraction Fluid #1 will be 4.93 $\pm$ 0.05. No standardization of acetic acid can be done - See Section 5.7.1 of Method in Federal Register 6-29-90.
   3. The pH of Extraction Fluid #2 will be 2.88 $\pm$ 0.05. Standardization of acetic acid is not mandatory but will be done for informational purposes (Optional) and will be compared to theoretical value of 5.7 mL. glacial acetic acid diluted to 1 liter. Titration of acetic acid normality can not be used for contract compliance purposes if correct pH value is obtained (2.88).

   ITEMS "a" THROUGH "e" AND ITEM "g" MUST BE A PART OF FORM I REPORT.

Analysis of TCLP extracts will be done to determine compliance with Regulatory Levels using minimum sample aliquot volumes necessary for this purpose.

Determination	Sample Aliquot (mls)	**Final Volume Taken for SOW Analysis After Dilution of** Sample Aliquot (ml)
Semi-Volatiles	1,000 for base/Neutral Extraction	1,000 (no dilution)
	50 for Acid Extraction	1,000

Sample aliquot sizes are to be minimized, as above, to alleviate interferences from acetic acid/acetate buffer, to provide CRQLs that are 10 - 20% of Regulatory Levels, and to expand the working concentration range of the methods.

ATTACHMENT I (con't)

Sample extraction will be done as initial Base/Neutral Extraction at pH > 12, using 1-liter of TCLP extract. Base/Neutral surrogates are expected to be extracted in this step. After base neutral extraction, 50 mls of basic aqueous solution will be diluted to 1000 mls and extracted under acid conditions pH 1.9-2.0 with careful use of sulfuric acid addition so as not to sulfonate phenol. pH shall not be less than pH 1.9. Extracts of each will be concentrated to 1.0 ml per SOW 2/88 with separate analysis of each extract for all required Table I TCLP constituents and surrogates. If a constituent is found in each extract, Form I values will be the sum of the 2 concentrations. Note that only one Form I is required for each sample.

1. 2,4-Dinitrotoluene and hexachlorobenze are limiting at 100 ug/l for Base-Neutral Extraction.

2. 2,4,6 Trichlorophenol is limiting at 2,000 ug/l for Acid Extraction.

Analysis of TCLP extract regulated constituents will require use of a daily continuing calibration standard for pyridine * besides the organics SOW calibration standards. Analysis of TCLP extracts will also be done for the Deferred TCLP constituent listed below:

1,2 Dichlorobenzene
Phenol

* Initial 5 Point Cal. curves are not necessary for pyridine but daily continuing calibration standard is required to be added to SOW 2/88 or OLM01.

Deferred TCLP Constituent 2,3,4,6-Tetrachlorophenl is not required.

All constituents of Table 1 and all surrogates of SOW 2/88 or OLM01 are required to be determined and reported for TCLP extracts. Remaining TCL's and TIC's of SOW 2/88 or OLM01 are not required. A matrix spike (MS)/matrix spike duplicate (MSD) for all constituents in Table 1 will be prepared and determined using one of the TCLP sediment extracts.

MS/MSD results are advisory and are used for calculation purposes. Surrogate recoveries are mandatory per requirements of SOW 2/88 or OLM01 for semi-volatiles.

TABLE 1
SEMI-VOLATILE TCLP CONSTITUENTS TO BE DETERMINED BY METHOD 1311, TCLP REGULATORY LEVELS, SAMPLE ALIQUOT VOLUMES TO BE USED, AND MS/MSD LEVELS AND CRQLS TO BE USED IN FINAL DILUTED SAMPLE ALIQUOTS[1]

Semi-Volatiles (SOW 2/88)(for initial Base/Neutral Extraction) (1,000 mL Sample Aliquot volume)

Contaminant	Regulatory Level (ug/L)	MS/MSD Level in Final Aliquot Dilution (ug/L)	CRQL in Final Aliquot Dilution (See Note 2) (ug/L)
bis(2-chlorethyl)ether	50*	100	SOW 2/88/OLM01
1,2-dichlorobenzene*	4,300*	100	SOW 2/88/OLM01
1,4-dichlorobenzene	7,600	100	SOW 2/88/OLM01
2,4-dinitrotoluene	100	100	SOW 2/88/OLM01
Hexachlorobenzene	100	100	SOW 2/88/OLM01
Hexchloro-1,3-butadiene	500	100	SOW 2/88/OLM01
Hexachlorethane	3,000	100	SOW 2/88/OLM01
Nitrobenzene	2,000	100	SOW 2/88/OLM01
Pyridine(1)	5,000	100-200	50
o-cresol	200,000	4,000[2]	SOW 2/88/OLM01
m&p-cresol	200,000	4,000[2]	SOW 2/88/OLM01
pentachlorophenol	100,000	4,000[2]	SOW 2/88/OLM01
phenol*	74,000	4,000[2]	SOW 2/88/OLM01
2,4,5-trichlorophenol	400,000	4,000[2]	SOW 2/88/OLM01
2,4,6-trichlorophenol	2,000	4,000[2]	SOW 2/88/OLM01

*-Deferred TCLP constituent with proposed regulatory level of 6/13/86.

(1) - Pyridine requires continuing calibration standard that is not part of SOW 2/88.

(2) After Base/Neutral Extraction, 50 mls of water is diluted to 1,000 mls and extracted as Acid Extraction at pH 1.9 to 2.0. Acid surrogates will have to be added at 2,000 ug/l concentrations.

Note 1: TCLP extraction of 6/29/90 requires correction of constituent values for matrix spike recoveries. See Section 8.2 of Method 1311 of 6/29/90. The average MS/MSD recoveries developed for one (1) of the soil extracts will be applied to all soil extracts. The initial MS/MSD determination level is selected within the analytical range of the SOW 2/88 or OLM01 to minimize or eliminate the need for MS concentrations over 3 order magnitude. It is not expected that the samples will provide TCLP values that will exceed regulatory levels; however there is a finite chance that this will occur. If any one TCLP analyte in an extract exceeds regulatory levels, the extract reanalysis is unnecessary using a Regulatory Matrix Spike concentration (see Section 8.2 of Method 1311).

If the concentration of the analyte after correction for the matrix spike recovery is > 10% and less than the regulatory level, the TCLP extract must be reextracted using a smaller aliquot and spiked at the regulatory level such that the native analyte is at approximately the regulatory level.

Reanalysis of the unspiked sample and the matrix spiked sample extracts will be done at the same dilution (see Section 8.2.2).

Note 2: For TCLP extract analysis detection limits, using SOW 2/88 or OLM01 (Organics), the SOW criteria are used; however, if these cannot be achieved due to matrix interferences, or any other valid reason detection finally reported should if at all possible be 10% or less, of the constituent's or contaminant's regulatory level. The case narrative for the extract analyses should discuss reason(s) why required detection limits cannot be met and the relation of final detection limits reported to regulatory level.

SAS Number

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490 or FTS/557-2490

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V
B. RSCC Representative: Jan Pels
C. Telephone Number: (312) 353-2720
D. Date of Request: March 1993
E. Site Name: Southeast Rockford Phase II Remedial Investigation

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of soils/sediments/solids by Toxicity Characteristic Leaching Procedure (TCLP) -- Method 1311 of Federal Register June 29, 1990 for regulated volatile TC Constituents, and certain deferred volatile TC constituents. Analysis of TC extracts will be done by organic SOW 2/88 or OLM01. Use a minimum sample aliquot to determine compliance with TCLP regulatory levels. TCLP extraction must be done within 14 days of date of sample collection. Volatile concentrations and extraction information will be reported on Form I's; also, volatile concentrations corrected for average MS/MSD recovery will be reported on separate Form Is for each sample.

2. Definition and number of work units involved specify whether whole samples or fractions; whether aqueous or soil and sediments; and whether low, medium, or high concentration):
5 soil/sediment/solid samples (includes field duplicates)

3. Purposes of analysis (specify whether Superfund [Remedial or Enforcement], RCRA, NPDES, etc.):
Superfund Remedial

4. **Estimated date(s) of collection:**

April 1993

5. **Estimated date(s) and method of shipment:**

Daily by overnight carrier

6. **Number of days analysis and data required after laboratory receipt of samples:** 30
Samples must be extracted within 14 days of collection; TCLP extracts must be analyzed within 14 days of extraction. Data package due July 1993

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**
Method 1311 of Federal Register - June 29, 1990 for extraction. CLP SOW 2/88 or OLM01 for analysis of extracts with modifications of Attachment I and Table 1.
Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

8. **Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):** Soils will be collected in 1 quart Bottle for organics. Case narrative should discuss any sample preparation problems. If interstitial water is present when the sample arrives at the laboratory, please premix water with soil prior to initiation of Method 1311. Extra 1-quart Bottles will be provided for preparation of the MS/MSD. See Attachment I regarding standardization of hydrochloric acid solution (Section 5.3), and acid solution preparations.
CASE NARRATIVE MUST DISCUSS ANY SAMPLE PROBLEMS.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody documentation, etc.). If not completed, format of results will be left to program discretion.**
Attachment I and Method 1311 provide information required for extraction. Use SOW 2/88 or OLM01 for providing Table 1 constituents. Separate Form I's are to be provided for Table 1 constituents and for Table I constituents corrected for MS/MSD recoveries from TCLP extracts. Form I's will also include extraction information of Items "a" through "e" and Item "g" of Attachment I.
All procedures used must be clearly identified. All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

10. **Other (use additional sheets or attach supplementary information, as needed):**
Lab must submit all original field documentation (COCs, tags, SAS PLs, etc.) and originals of data to the Region in the time frame referenced in Section 6 of this SAS (analogues to a RAS-CSF).
All original sample tags, chain of custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in section 6 above.

11. **Name of sampling/shipping contact:** Yan Yan Ma
Phone: (312) 474-1313

TCLP Extraction will be done by Federal Register of June 29, 1990 including ZHE (attached). Samples will be soils, wet soils, sediments, or moist or dry solids; therefore, the filtration procedure (Section 7.1.1.7 of attached procedure) may produce iterstitial water. Also, any water collecting on top of sediment or soil is not to be discarded, but mixed with sample prior to filtration or % solid determination (Section 7.1.1). TCLP extracts may be a combination of liquid filtrate and solid TCLP extracts (see Sections 7.2.13.2 and 7.3.14) but will depend on the physical nature of the sediments collected. Also see Section 8 of SAS request. Particle size reduction is not expected to be necessary for soils and sediments. Sample preparation logs will be needed to record all required information of Method 1311 including (but not limited to):

a. Weight(s) of extracted samples (25g minimum aliquot size is required for 100% solids content) and volume of any filtrate (Sections 7.3.4 and 7.4.5.2).
b. Preliminary evaluations of percent (%) solids.
c. pH data
d. Dates of each preparation step, with associated weights and measured volumes.
e. pH value of final TCLP extract (Section 7.2.14)
f. Holding times (Section 7.4) are to be met and are to be counted from the date of collection.
g. Record HCl normality (Section 5.3) and acetic acid normality (Section 5.7.2) (SAS par. 8) and measured pH of Extraction fluid (Section 5.7.1 and 5.7.2). Record dates of each of required measurements.
h. The pH of Extraction Fluid #1 will be 4.93 $\pm$ 0.05. No standardization of acetic acid can be done - See Section 5.7.1 of Method in Federal Register 6-29-90.

ITEMS "a" THROUGH "e" AND ITEM "g" MUST BE A PART OF FORM I REPORT.

Analysis of TCLP extracts will be done to determine compliance with Regulatory Levels using minimum sample aliquot volumes necessary for this purpose.

Analysis of TCLP extracts will also be done for Deferred TCLP constituents listed below:

Acrylonitrile *(requires additional cont. cal. standard)
Carbon Disulfide
Methylene Chloride
1,1,1,2-Tetrachloroethane *(needs extra cont.cal. standard)
1,1,2,2-Tetrachoroethane
Toluene
1,1,1-Trichlorethane
1,1,2-Trichoroethane

* Initial -5 Point Cal. curves are not necessary for these 2 compounds but daily continuing calibration standards are required to be added to SOW 2/88.

Deferred TCLP Constituent of isobutanol is not required.

All volatile constituents of Table 1 and all surrogates of SOW 2/88 are required to be determined and reported for TCLP extracts. TIC's are not required. A matrix spike (MS)/matrix spike duplicate (MSD) for all constituents in Table 1 will be prepared and determined using one of the TCLP sediment extracts.

MS/MSD results are advisory and are used for calculation purposes. Surrogate recoveries are mandatory per requirements of SOW 2/88 or OLM01 for volatiles.

Determination	Sample Aliquot (mls)	Final Volume Taken for SOW Analysis After Dilution of Sample Aliquot (ml)
Volatiles (ZHE)	1	5

Sample aliquot sizes are to be minimized, as above, to alleviate interferences from acetic acid/acetate buffer, to provide CRQLs that are 10 - 20% of Regulatory Levels, and to expand the working concentration range of the methods.

TABLE 1 P. 1 OF 2

VOLATILE TCLP CONSTITUENTS TO BE DETERMINED BY METHOD 1311, TCLP REGULATORY LEVELS, SAMPLE ALIQUOT VOLUMES TO BE USED, AND MS/MSD LEVELS AND CRQLS TO BE USED IN FINAL DILUTED SAMPLE ALIQUOTS[1]

ontaminant	Regulatory Level (ug/L)	Sample Aliquot Volume ml	MS/MSD Level in Final Aliquot Dilution (ug/L)	CRQL in Final Aliquot Dilution (ug/L)
VOLATILES (SOW 2/88) (ZHE_)				(See Note 2)
crylonitrile	5,000[1]	1	100-200	100-200[1]
enzene	500	1	100	SOW 2/88/OLM01
arbon Disulfide*	14,000*	1	100	SOW 2/88/OLM01
arbon tetrachloride	500	1	100	SOW 2/88/OLM01
Chlorobenzene	100,000	1	100	SOW 2/88/OLM01
hloroform	6,000	1	100	SOW 2/88/OLM01
,2-Dichloroethane	500	1	100	SOW 2/88/OLM01
,1-Dichloroethene	700	1	100	SOW 2/88/OLM01
ethylene Chloride*	8,600*	1	100	SOW 2/88/OLM01
Methyl Ethyl Ketone	200,000	1	100-200	SOW 2/88/OLM01
,1,1,2 Tetrachloro-ethane*	10,000*[1]	1	100	100[1]
,1,2,2 Tetrachloro-ethane*	1,300*	1	100	SOW 2/88/OLM01
etrachlorethene	700	1	100	SOW 2/88/OLM01
Toluene*	14,000*	1	100	SOW 2/88/OLM01
,1,1-Trichloro-ethane*	25,000*	1	100	SOW 2/88/OLM01
,1,2-Trichloro-ethane*	1,200*	1	100	SOW 2/88/OLM01
richloroethene	500	1	100	SOW 2/88/OLM01
Vinyl Chloride	200	1	100	5-10

* -Deferred TCLP Constituent with Proposed Regulatory Level of 6/13/86.

1. Acrylonitrile and 1,1,1,2-Tetrachlorethane require continuing calibration

standard that is not part of SOW 2/88 or OLM01.
(See Attachment I for calibration requirements.)

Note 1: TCLP extraction of 6/29/90 requires correction of constituent values for matrix spike recoveries. See Section 8.2 of Method 1311 of 6/29/90. The average MS/MSD recoveries developed for one (1) of the soil extracts will be applied to all soil extracts. The initial MS/MSD determination level is selected within the analytical range of the SOW 2/88 or OLM01 to minimize or eliminate the need for MS concentrations over 3 orders of magnitude. It is not expected that the samples will provide TCLP values that will exceed regulatory levels; however there is a finite chance that this will occur. If any one TCLP analyte in an extract exceeds regulatory levels, the extract reanalysis is unnecessary using a Regulatory Matrix Spike concentration (see Section 8.2 of Method 1311).

If the concentration of the analyte after correction for the matrix spike recovery is > 10% of but less than the regulatory level, the TCLP extract must be reextracted using a smaller aliquot and spiked at the regulatory level such that the native analyte is at approximately the regulatory level.

Reanalysis of the unspiked sample and the matrix spiked sample extracts will be done at the same dilution (see Section 8.2.2).

Note 2: For TCLP extract analysis detection limits, using SOW 2/88 or OLM01 (Organics), the SOW criteria are used; however, if these cannot be achieved due to matrix interferences, or any other valid reason detection finally reported should if at all possible be 10% or less, of the constituent's or contaminant's regulatory level. The case narrative for the extract analyses should discuss reason(s) why required detection limits cannot be met and the relation of final detection limits reported to regulatory level.

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V
B. RSCC Representative: Jan Pels
C. Telephone Number: (312) 353-2720
D. Date of Request: March 1993
E. Site Name: Southeast Rockford Phase II Remedial Investigation

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delays in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of soils/sediments/solids by Toxicity Characteristic Leaching Procedure (TCLP) -- Method 1311 of Federal Register June 29, 1990 for the eight regulated metals. TCLP sample extracts must be prepared within 28 days of the date of sample collection for Hg and 180 days for all other metals. Use a minimum sample aliquot of TCLP extract to determine compliance with TCLP regulatory levels.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

5 Soil/sediment/solid samples (includes field duplicates) for analysis by TCLP -- Method 1311 of Federal register June 29, 1990 for the eight regulated metals, and for the 8 regulated metals corrected for average MS/MSD recovery.

3. Purposes of analysis (specify whether Superfund [Remedial or Enforcement], RCRA, NPDES, etc.):

Superfund Remedial

US4359

4. **Estimated date(s) of collection:**

- April 1993

5. **Estimated date(s) and method of shipment:**

- Daily by overnight courier

6. **Number of days analysis and data required after laboratory receipt of samples:**

Samples must be extracted within 28 days of collection for Hg and within 180 days for all other metals; TCLP extracts must be analyzed within 28 days of extraction for Hg.

Data package due July 1993

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

Method 1311 of Federal Register - June 29, 1990 for extraction. CLP SOW 7/88 or ILM01 for analysis of extracts with modifications of Attachment I and Table 1.

Note: Laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

8. **Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

See Attachment I and Table 1. Soils will be collected in 1-liter wide-mouth glass jars for metals. Samplers are instructed to add only soil. If interstitial water is present on arrival the laboratory, please remix water with soil prior to initiation of Method 1311. Standardize the acetic acid solutions (Section 5.7.2), and the 1N HC1 (Section 5.3) by titrating with standard 0.1N NaOH before use. Must be within +/-5% of required value. Analysis of diluted TCLP extract shall be done using SOW 7/88 or ILM01 and QC requirements of Attachment II.

CASE NARRATIVE MUST DISCUSS ANY SAMPLE PROBLEMS.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain of Custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Attachment I and Method 1311 provide information required for extraction. Use SOW 7/88 or ILM01 for providing Table 1 constituents. Separate Form I's are to be provided for Table 1 constituents and for Table 1 constituents corrected for average MS/MSD recoveries from TCLP extracts. Form I's will also include extraction information of Items "a" through "e" and Item "g" of Attachment I.

All procedures used must be clearly identified. All original raw data, forms, calculation worksheets, instrument read-outs, preparation forms, internal sample and/or extract chain of custody forms, strip charts, and copies of pages from preparation and analysis logbooks shall be submitted. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

All records of analysis and calculations shall be legible and sufficient to recalculate all sample concentrations and QA audit results. QC reference samples or initial calibration standards shall be identified as to source, lot number, and sample number.

10. **Other (use additional sheets or attach supplementary information, as needed):**

All original sample tags, chain of custody forms, SAS packing lists, airbills, and any other original receiving or transmittal forms, or copies of receiving logbook pages, pertaining to this SAS shall be submitted to the Region within the time frame listed in section 6 above.

11. **Name of sampling/shipping contact:** - Yan Yan Ma

Phone: - (312) 474-1313

I. DATA REQUIREMENTS

Parameter	Detection Limit	Duplicate Precision Desired
See Table I - all ICP measurements of SOW 7/88 or ILM01 are to be included but remaining TAL's need not be reported.	See Table I.	+/- 25% difference (advisory for TAL's)

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audit	Frequency of Audits	Limits (% or Conc.)
TCLP Extraction		
Prep. Blank for Extract Fluid #1 (see 7.1.4.4 of Method 1311)	Each set of solid samples prepared.	< 5% of regulatory levels of Table I. Discuss in case narrative if larger than CRDL's of SOW.
Prep. Blank for Extract Fluid #2 (if necessary)	Same as above.	Same as above.
Analysis of TCLP Extracts		
Preparation Blank for TCLP Extract Determinations.	Per appropriate SOW and set-up with each TCLP extract batch.	CRDL of Appropriate SOW for Table I constituents.
MS/MSD (see Table I)	See Table I (required for inorganics) 1 for each set of 8 sample extracts.	Advisory - used to correct TCLP values recovery. See Note 1 of Table I. RPD < = 20% (MS/MSD)
All other QC audits per SOW 7/88 or ILM01.	per SOW 7/88 or ILM01.	per SOW 7/88 or per ILM01

Reference standards approved by the EPA under the CRADA (Cooperative Research and Development Agreement) program. These materials must bear the words "USEPA Certified" or "A2LA Certified".

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact SMO and Region V. Reanalysis of TCLP extracts may be necessary per requirements of Note 1 of Table I.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

TCLP Extraction will be done by Federal Register of June 29, 1990 (attached) including bottle extraction for metals. Samples will be wet soils or sediments; therefore, the filtration procedure (Section 7.1.1.7 of attached procedure) may produce interstial water. Also any water collecting on top of sediment or soil is not to be discarded, but mixed with sample prior to filtration or % solid determination (Section 7.1.1). TCLP Extracts may be a combination of liquid filtrate and solid TCLP extracts (see Sections 7.2.13.2 and 7.3.14) but will depend on the physical nature of the soils collected. Particle size reduction is not expected to be necessary for these soils. Sample preparation logs will be needed to record all required information of Method 1311 *including (but not limited to):*

a. Weight(s) of extracted samples (100g minimum aliquot size is required for 100% solids content) and volume of any filtrate (Sections 7.2.2 and 7.2.5).
b. *Preliminary evaluations of percent (%) solids.*
c. pH data for selection of Extraction Fluid #1 or #2 (Section 7.1.4.2)
d. Dates of each preparation step, with associated weights and measured volumes.
e. pH value of final TCLP extract (Section 7.2.14)
f. *Holding times (Section 7.4) are to be met and are to be counted from the date of collection.*
g. Record HCl normality (Section 5.3) and acetic acid normality (Section 5.7.2) (SAS par. 8) and measured pH of Extraction fluids (Section 5.7.1 and 5.7.2). Record dates of each of required measurements.
h. Standardization of Hydrochloric acid and acetic acid for Extraction Fluid #1 and Extraction Fluid #2.
1. The 1 N HCl can be and will be standardized to 1.0 N HCl $\pm$ 5%.
2. The pH of Extraction Fluid #1 will be 4.93 $\pm$ 0.05. No standardization of acetic acid can be done - See Section 5.7.1 of Method in Federal Register 6-29-90.
3. The pH of Extraction Fluid #2 will be 2.88 $\pm$ 0.05. Standardization of acetic acid is not mandatory but will be done for informational purposes (Optional) and will be compared to theoretical value of 5.7 mL. glacial acetic acid diluted to 1 liter. Titration of acetic acid normality can not be used for contract compliance purposes if correct pH value is obtained (2.88). TCLP combined extract aliquots will be acidified for subsequent metals analysis (Section 7.2.14).

ITEMS "a" THROUGH "e" AND ITEM "g" MUST BE A PART OF FORM I REPORT.

Analysis of TCLP extracts will be done to determine compliance with Regulatory Levels using minimum sample aliquot volumes necessary for this purpose.

Determination (ml)	Sample Aliquot (mls)	Final Volume Taken for SOW Analysis After Dilution of Sample Aliquot
Metals (ICP)	10	100
Metals (GFAA)	10	100
Hg (CVAA)	5	100

Sample aliquot sizes are to be minimized, as above, to alleviate interferences from acetic acid/acetate buffer, to provide CRQLs that are 10 - 20% of Regulatory Levels, and to expand the working concentration range of the test procedures.

All constituents of Table 1 are required to be determined and reported for TCLP extracts. Remaining TALs of 7/88 or ILM01 are not required, except that all ICP emission spectroscopy measurements required by SOW 7/88 or ILM01 are to be made and included in raw data. A matrix spike (MS)/matrix spike duplicate (MSD) for all constituents in Table 1 will be prepared and determined using one of the TCLP soil extracts. The same extract need not be used for all analyses (ICP, GFAA, or CVA).

MS/MSD results are advisory and used for calculation purposes.

TABLE 1
TCLP CONSTITUENTS TO BE DETERMINED BY METHOD 1311,
TCLP REGULATORY LEVELS, SAMPLE ALIQUOT VOLUMES TO BE USED,
AND MS/MSD LEVELS AND CRQLS TO BE USED
FINAL DILUTED SAMPLE ALIQUOTS (100 ml)[1]

Contaminant Metals (SOW 7/88 or ILM01)	Regulatory Level (ug/L)	Sample Aliquot Volume (ml)	MS/MSD Level in Final Aliquot Dilution (ug/L)	CRQL in Final Aliquot Dilution
As (GFAA)	5,000	10	500	50
Ba (ICP)	100,000	10	10,000	1,000
Cd (ICP)	1,000	10	10	10
Cr (ICP)	5,000	10	50	50
Pb (ICP or GFAA)	5,000	10	50	50
Hg (CVAA)	200	5	0.5	0.5
Se (GFAA)	1,000	10	10	10
Ag (ICP)	5,000	10	50	50

Note 1: TCLP Extraction of June 29, 1990 requires correction of constituent values for matrix spike recoveries. See Section 8.2 of Method 1311 of Federal Register June 29, 1990.

The average MS/MSD recovery developed for 1 of the soil extracts will be applied to all of the soil extracts. It is not expected that the samples will provide TCLP values that will exceed Regulatory Levels; however, there is a finite chance that this will occur.

If any one TCLP analyte in an extract exceeds Regulatory Levels, the extracts reanalysis is unnecessary using a Regulatory Matrix Spike concentration (see Section 8.2 of Method 1311).

If the concentration of the analyte after correction for the matrix spike recovery is > 10% of but less than the regulatory level, the TCLP extract must be reextracted using a smaller aliquot and spiked at the regulatory level such that the native analyte is at approximately the regulatory level.

If sample concentrations exceed the calibration range, sample must be diluted to fall within the calibration range.

Environmental Protection Agency
Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

TOC IN SOIL

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

EPA Region/Client: Region V

RSCC Representative: Jan Pels

Telephone Number: (312) 353-2720

Date of Request: March 1993

Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Determination of organic carbon (%) in soil (air-dried-all screened through 100 or 140 mesh). Applicable concentration 0.1% to 10.0 % or more. Detailed information must be provided with Case Narrative for test procedures, instrumentation/apparatus and QC used. See Attachment 1.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

33 samples - 30 investigative, 3 field duplicates for low concentration sediment samples.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund, ~~Enforcement~~ REMEDIAL

US4365 2/15/91 Rev. 2.0

Estimated date(s) of collection: April 1993

Estimated date(s) and method of shipment: overnight courier

Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days of receipt of last sample.

Analytical protocol required (attach copy if other than a protocol currently used in this program):

See Attachment 7

Also see Attachment A.

Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

See Attachment 8

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

See Attachment 9

Also see Attachment B

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
Organic Carbon % in Soil	0.10%; report actual detection limit if smaller	± 20%; on duplicate sample results

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)
1 Prep. Blanks	1 in every 10 samples or at least twice	≤0.1%
2 Duplicate Samples	1 in 5 samples	<20% RPD in differences of duplicate sample results, or <0.2% differences at small concentrations.
3 Positive Control (to be determined by the lab)	1 in 10 samples	88-115% recovery
4 Instrument Calibration Checks and Calibration Blanks (if appropriate)	1 in 10 or fewer samples	90-110% recovery for calibration check, and <0.1% total carbon for assumed routine sample weight

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact Region V RSCC.

Jan Pels 312/ 353-2720

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT I

Determination of organic carbon(%) in soil, using sub-aliquots of air-dried soil, passed through a 100 mesh to 140 mesh screen. All of the sub-aliquot must pass the screen. Applicable organic carbon concentration range of interest is 0.1% to 10% (or larger) in soil, (dry weight basis). Laboratory may report lower concentration values.

Test procedures used for determining soil shall be the 1) dry combustion (resistence furnace), 2) Dry combustion (induction furnace), 3) Dry combusiton (automated methods), or 4) Wet combustion (combustion train) methods of analysis specified by Table 29-1 of "Methods of Soil Analyses," Part 2 - Chemical and Microbiological properties, 2nd ed., 1982, American Society of Agronomy, and Soil Science Society of America, Madison, Wisconsin. Copies of this copyrighted material are not being provided, because laboratories doing organic carbon analysis of soil should already have it.

Any automated dry combustion test procedure used must provide results consistent with the other 3 methodologies and must be consistent with the requirements of Chapter 29, Sections 29-1, 29-2, and 29-3, "Methods of Soil Analysis" (MSA) Part II, 2nd ed., as appropriate. Soils can be calcerous or noncalcerous soils, with varying amounts of organic carbon. Soils determined may be subsurface as well as surface soils. If peat or muck soils are ever encountered, the laboratory will provide with the case narrative, limitations of any sample results and any solutions to problems encountered. This is also true for any other problem sample types encountered.

The laboratory, providing organic carbon analysis data, will provide information with the case narrative concerning methodology, instrumentation, and specific QA practices used for the set of soils tested. Requested information is detailed in items #8, and #9 of this SAS.

ATTACHMENT 7
Analytical Methods - Organic Carbon in Soil

7a. Sample Preparation: Representative sub-aliquot of air-dried soil (see % solids SAS) screened through 100 or 140 mesh as appropriate. All of the sub-aliquot must pass this screen.

b. Test for Presence of Inorganic Carbon, MSA, Part II, Section 29-3.3.1. Place finely ground soil on a spot plate, and moisten with a few drops of water. Add 4 N HCl dropwise to the wetted sample and observe any effervescence. Allow sufficient time for dolomite to react (~5 min). If inorganic carbon is absent proceed with Total Carbon in items #7c, or 7d below. If inorganic carbon is present, or the test is not definitive, proceed with tiems #7e, & #7f prior to Total Carbon measurements of Item #7c or #7d.

c. Total Carbon (Dry Combustion), MSA, Part II, Section 29-2.2.2. Use this as a guide for instrumental specifications. Instrument must test solid sample directly. Illustrative examples of this methodology are:

1) Total Carbon (Dry Combustion - Medium Temperature Resistance Furnace), MSA, Part II, Section 29-2.2.3.

2) Total Carbon (Dry Combusiton - High Temperature Induction Furnace), MSA, Part II, Section 29-2.2.4.

3) Total Carbon (Dry Combusiton - Other Instrumental Methods), MSA, Part II, Section 29-2.2.5. Any other instrumentation such as this must be justified and provide results as precise and accurate as the results from Sections 29-2.2.3, and 29-2.2.4.

d. Total Carbon (Wet Digestion), MSA, part II, Section 29-2.3.2 Soil digested in 60:40 mixture of sulfuric acid and phosphoric acid (containing $K_2Cr_2O_7$). CO_2 evolved is absorbed and weighed, or absorbed in standard base and titrated.

1) Specific examples are found in MSA, Part II, Figure 29-2, Figure 29-3, and Section 29-2.3.3.

e. Pretreatment prior to Dry Combustion, MSA, Part II, Section 29-3.3.3. Inorganic carbon is removed by treating sample in a combustion boat, with 5% sulfurous acid (H_2SO_3). After several hours, remove the excess H_2SO_3 by leaving overnight in an evacuated dessicator. Read citation for further details.

f. Pretreatment prior to Wet Digestion, MSA, Part II, Seciton 29-3.3.2. Inorganic carbon is removed by sulfuric acid - ferrous sulfate reagent in apparatus used for total carbon (Wet Digestion) prior to Total Carbon measurement. See citation for further details.

- (cont. next page) -

g. Use only the methods specified above or obtain approval of CPMS, CRL prior to use of other method. Test procedure description, and description of specific measurement principles including equivalency to each of the 10 items of Figure 29-1 of MSA, part II and sample pretreatmenst of Section 29-3, MSA, Part II.

h. Laboratory performing Total Carbon determinations must use and have a recognized procedure for removal of any inorganic carbon in sample.

A varirty of apparatus, instrumentation, sample preparation systems and read-outs can be used. It is the responsibility of the laboratory to provide appropriate QC audits and QC data with each set of samples tested.

If instrumentation requires calibration, provide calibration curve, including zero concentration standard and preparation blanks. Provide positive control (a test sample prepared independently from calibration standards) that provides a measure of accuracy of system. This should be done for all systems including grammetric read-outs.

When using an automated analytical system, the following calibration procedure must be used and documented in the raw data and on summary sheets:

1. Initialize and standardize the curve.

2. Assay the zero standard and the four calibration standards.

3. If the calibration standard concentrations are [redacted] ± 5% of the 'true value' for the midrange and high level standards and ± 10% for the low to midrange standards, the curve can be used.

4. If the standards are outside of the windows in #3 above, the lab must reinitialize and standardize the curve, and perform steps 2-3 again. If the lab cannot meet the criteria specified above, contact SMO immediately.

If the EPA QC check sample results (or the independently prepared positive control sample results) are out of control analyses must be halted, corrective action must be taken and samples must be rerun.

ATTACHMENT 9
Analytical Results Required

As part of Case Narrative, attach description of test procedure and instrumentation used for measurement of Total C and removal of any Inorganic C. Test procdure description must include sufficient information that the nature of specific analytical result deliverables can be determined including QC audits. In Case Narrative, discuss any problem type samples (including peat or muck soils), limitations on any sample results, and soultion taken to resolve any problems. A sample preparation log will be provided, as appropriate.

Bench record tabulating any order of any sample weights and tare weights of absorbed CO_2, instrument calibrations, blanks, QA audits, etc., must be provided along with copies of any worksheets used to calculate results. Include copies of any instrument readouts. All must be legible. Report results as % organic Carbon on a dry weight basis (103-105°), using guidance in % solids SAS. Note that the analysis is performed on an air dried aliquot but that results must be reported on a dry weight basis (103-105°).

...nmental Protection Agency
Management Office
818, Alexandria, Virginia 22313
...ONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: March, 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of drinking water/ residential wells for volatiles with low quantitation limits

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples – 25 investigative, 3 field duplicates, 3 field blanks for low concentration drinking water.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund REMEDIAL

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight couriers

6. Number of days analysis and data required after laboratory receipt of samples: Data package should be received in the Region within 14 days on a Sample Delivery Group basis. Holding times for each fraction are defined in the Method 6/91.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Analysis per the Superfund Analytical Methods for Low Concentration Water For Organic Analysis, 6/91.

Also see Attachment A

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): Notify the Region if dilutions are requir

1. Occasionally, monitoring well samples may be sent; in the case where higher levels are suspected, the lab will receive notification when scheduling and the samples will be identified by field samplers on the Traffic Report form or the Chain of Custody form. The lab w need to screen these samples to determine the minimum dilution required; the Region r be notified when dilutions are required for further instructions regarding reporting

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

As per 6/91 Method. Also see Attachment B

The lab must notify SMO if any reanalyses or reextractions need to be performed outside of holding times (i.e., due to surrogate recovery pr etc.) so the Region can determine whether to submit the original analy

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

Rev. 9-2
12/91

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Volatile Organics Only As per Method 6/91	As per 6/91	As per 6/91

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)
As per 6/91	As per 6/91	As per 6/91
Storage Blank as per 6/91 SOW	As per 6/91 SOW	As per 6/91 SOW

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

As per Method 6/91, Call SMO.

Please return this request to the Sample Management Office as soon as possible to expedi processing of your request for special analytical services. Should you have any questic or need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

(Method 6/91)

OLCO1.0 SUMMARY

The following is a summary of the major differences in the new Organic Low Concentration Statement of Work as compared to the Organic Multimedia Statement of Work. Data turnaround is 14 days, therefore, the Sample Delivery Group (SDG) is on a 7 day basis. A Complete SDG File (CSF) Inventory requirement replaces the file purge requirement. All original sample documentation, including sample tags, custody forms, airbills, etc., will be shipped directly to the Region with a check off sheet and the complete original data package in 14 days. New forms include the Sample Log-in Sheet (DC-1) and the Document Inventory Sheet (DC-2). The diskette deliverable applies. Performance Evaluation Samples (PES) will be sent with each shipment of samples and will be analyzed on an SDG basis; these samples will be supplied by the EPA and action can be taken if the results score is <75%. No matrix spike/matrix spike duplicate analyses are required.

VOLATILE ORGANIC COMPOUND ANALYSIS

This fraction's Target Compound List (TCL) contains 40 compounds plus 10 Tentitively Identified Compounds (TIC).

New BFB tuning criteria and procedures are included.

A 7 day holding time is required. This will be changed to a 10 day holding time in the near future; all VOA vials should be preserved in the field to a pH < 2.

Sample analysis volume is 25 ml.

A Laboratory Control Sample (LCS) composed of 12 compounds is analyzed with each SDG; if LCS recovery limits are not met, re-analysis of all samples in the SDG is required.

One surrogate compound, BFB, is used and has 80-120% recovery limits.

Three internal standards (IS) are used: 1,4-difluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4; $\pm$ 40% area differences and RTs of $\pm$ 0.33 min (20 sec) are allowed.

Initial calibration is at 1,2,5,10,and 25 ppb with a continuing calibration at 5 ppb.

Response factors, % relative standard deviation (RSD), and % difference (D) for response factors have control limits that are compound specific for 28/40 of the compounds, with an exemption for 2 compounds out of control with a maximum % RSD or % D of 40; a minimum RF of 0.01 is required for the remaining 12 compounds.

Bromochloromethane, formerly an IS, is now a target compound.

Volatiles (continued)

Cis-1,2-dichloroethene and trans-1,2-dichloroethene are reported separately.

Vinyl acetate has been removed from the target compound list.

Added to the target list are: 1,2-dibromoethane, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, and 1,2-dibromo-3-chloropropane.

Contract required quantitation limits (CRQL) are 1 ppb for all compounds except for methylene chloride at 2 ppb, and acetone, 2-butanone, 4-methyl-2-pentanone and 2-hexanone at 5 ppb.

Subambient temperature GC programming is required.

Blank concentrations of all target compounds must be < CRQL.
Blank TIC concentrations must be < 2.0 ppb.

TABLE 1 (ALL UNITS ARE MICROGRAMS/LITER)

PARAMETER	CAS #	QUANTITATION LIMITS
BENZENE	71-43-2	1.5
BROMODICHLOROMETHANE	75-27-4	1.5
BROMOFORM	75-25-2	1.5
BROMOMETHANE	74-83-9	10
CARBON TETRACHLORIDE	56-23-5	1.5
CHLOROBENZENE	108-90-7	1.5
CHLOROETHANE	75-00-3	1.5
CHLOROFORM	67-66-3	1.5
CHLOROMETHANE	74-87-3	10
DIBROMOCHLOROMETHANE	124-48-1	1.5
1,1-DICHLOROETHANE	75-34-3	1.5
1,2-DICHLOROETHANE	107-06-2	1.5
1,1-DICHLOROETHENE	75-35-4	1.5
TOTAL 1,2-DICHLOROETHENES	156-60-5	1.5
1,2-DICHLOROPROPANE	78-87-5	1.5
cis-1,3-DICHLOPROPROPENE	10061-01-5	2
trans-1,3-DICHLOROPROPENE	10061-02-6	1
ETHYL BENZENE	100-41-4	1.5
METHYLENE CHLORIDE (*)	75-09-2	1
1,1,2,2-TETRACHLOROETHANE	79-34-5	1.5
TETRACHLOROETHENE	127-18-4	1.5
TOLUENE (*)	108-88-3	1.5
1,1,1-TRICHLOROETHANE	71-55-6	1.5
1,1,2-TRICHLOROETHANE	79-00-5	1.5
TRICHLOROETHENE	79-01-6	1.5
VINYL CHLORIDE	75-01-4	10
ACETONE (*)	67-64-1	75
CARBON DISULFIDE	75-15-0	3
2-BUTANONE	78-93-3	(50)
4-METHYL-2-PENTANONE	108-10-1	(3)
2-HEXANONE	519-78-6	(50)
STYRENE	100-42-5	1
o-XYLENE **	95-47-6	
p-XYLENE **	106-42-3	2.5

* COMMON LABORATORY SOLVENT
BLANK LIMIT IS 5x METHOD DETECTION LIMIT

** THE o-XYLENE AND p-XYLENE ARE REPORTED AS A TOTAL OF THE TWO

9/002__-0-6/87 Alkalinity (H_2O) 6/29/87

.S. Environmental Protection Agency
LP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES Approved For Scheduling
Client Request

| X | Regional Transmittal | | Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: JAN PELS

C. Telephone Number: 312-353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for alkalinity in waters (surface waters, groundwaters, drinking waters, leachates, etc.). Samples will be unfiltered. Reports are reported as mg/l $CaCO_3$.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blank.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4379

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) Alkalinity EPA Method 310.1 (Titrimetric, ph 4.5) 2) Standard Methods, 16th Edition, Method 403 4c and 4d.

Samples will be stored at 4°C until analysis and validation of results.

Also see Attachment A

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples holding time should not exceed 14 days from date of collection. Use potentiometric titration to pH 4.5 for alkalinity > 20 mg/l as $CaCO_3$. For concentrations <20 mg/l, use EPA Method 310.1 (Section 6.3) or Standard Methods, Method 403 4d. Do not use titrant volumes greater than 50ml. Obtain approval of CPMS, CRL prior to use of any other method.

Use Na_2CO_3 to standardize titrant. Standardize the pH meter and the titrant each day.

Standardize the pH meter using at least two buffers which bracket the end point.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The Test procedure used will be clearly identified. Bench records tabulating the order of analysis including pH meter calibration, titrant standardization, lab blanks, samples, lab control standards, duplicates, etc., with resulting titrant volumes or readouts will be provided along with calculation worksheets. All records will be legible and sufficient to recalculate all sample concentrations and QA audit results. Report method of titrant standardization.
EPA QC Reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used. Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (+% or Conc.)
Alkalinity	2 mg/l for low level	± 2 mg/l for Conc. < 20 mg/l $CaCO_3$
	20 mg/l for high level	± 10% for Conc. > 20 mg/l
NOTE: These are minimum requirements. Report actual detection limits used based on allowable methodologies.		

I. QC REQUIREMENTS - Do not use designated field blanks for QA audits. The QA audits below will be done for each group of low-level and high-level akalinity determinations.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
lab blank	at least 1 per group of 10 or fewer samples	<10 mg/l for high-level samples tested. <2 mg/l< for low-level samples tested.
lab duplicate	at least 1 per group of 10 or fewer samples	± 10% or ± 2 mg/l
lab control sample	1 per sample set	90-110% recovery.
1 set of EPA QC mineral reference samples		

I. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanlyze samples.

Contact ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.~~ Sample Management Office

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions need any assistance, please call the Sample Management Office.

US4381

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/014__-0-6/87 Nitrate/nitrite 6/29/87

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

APPROVED FOR SCHEDULING

SPECIAL ANALYTICAL SERVICES
Client Request _____ _____

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: JAN PELS

C. Telephone Number: 353-2720

D. Date of Request: March 1993

E. Site Name: ~~SOE~~ SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of nitrate plus nitrite (as mg/l N) in water (surface water ground water, drinking water, leachates, etc.)

Samples will be unfiltered.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blanks.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4383

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: DAILY BY OVERNIGHT CARRIER

6. Number of days analysis and data required after laboratory receipt of samples: Laboratory should report results within 30 days of receipt of samples

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1) EPA Method 353.1 (colorimetric, automated hydrazine reduction).
2) EPA Method 353.2 (colorimetric, automated cadmium reduction).
3) EPA Method 353.3 (colorimetric, manual cadmium reduction).
For all methods:
Samples will be stored at 4°C until analysis and validation of results. Samples will be preserved in the field with sulfuric acid (1 ml/l) to pH<2. The analytical working range shall not exceed 0.1 to 10.0 mg/l N.

For Methods 353.2 or 353.3: If more than one reduction column is used separate calibrations, QA audits, and records are required for each column. The column used must be identified for each analytical result. Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
Analyze the samples within 28 days after collection. Check the sample pH (wide range pH paper is acceptable). If the pH>2 contact CPMS, CRL for instructions. Use only the methods specified in item 7. Obtain approval of CPMS, CRL before using any other method.
For Methods 353.2 and 353.3: After checking the pH it is recommended that the laboratory check for residual chlorine (or oxidizing reagents) and sulfide using test strips such as starch iodide and lead acetate papers. Contact CPMS, CRL if these interferences are present; however, the laboratory must remove these interferences prior to analysis. The laboratory must also minimize interferences due to metals in order to prolong column life. (See Section 7.1.2 of method 353.3) It is suggested that the laboratory may dilute samples up to ten-fold prior to analysis (Section 7.4 of Method 353.3) provided that the final analytical working range does not exceed 0.1 to 10.0 mg/l N.
For all methods: Neutralize samples to pH 5-9 (or to phenolphthalein color end-point) prior to analysis. Dilute and reanalize the neutralized samples if the concentrations exceed that of the highest standard. Use at least five calibration standards (including a zero standard). Prepare the lab blank using 1 ml of H_2SO_4/l. Neutralize and analyze it like a sample.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:
The test procedure used must be clearly identified. Bench records tabulating the order of calibration standards, lab control standards, lab blanks, samples, spikes, duplicates, etc., with resulting absorbances or concentration readouts will be provided. Worksheets used to calculate results will be included. Any sample treatment to remove interferences will be documented. The laboratory shall submit photo-copies of the instrument readout (strip-charts, printer tapes, etc.) All records of analysis and calculations must be legible and sufficient to recalculate all concentrations. Results are to be reported as mg N/l.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used. Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as need
See Attachment C. US4384

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312- 474- 1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Nitrate + Nitrite	0.10 mg/l as N	Duplicate results must be within 10% for concentrations >1mg/l or within 0.1 mg/l for concentrations ≤ 1mg/l Results will be reported to the nearest 0.1 mg/l for conc. less than 1.0 mg/l and to 2 significant figures for conc. exceeding 1 mg/l-N.

Note: These are minimum requirements. Report actual detection limits used based on allowable methodology options.

I. QC REQUIREMENTS - Do not use any designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	1 per group of 10 or fewer samples	±(10% - or 0.1 0 mg/l)
Lab Blank (1ml/l H_2SO_4)	2 per sample set	<0.1 mg/l
Calibration verification standard	1 per group of 10 or fewer samples and at end of run	90% - 110%
Calibration blank	1 per group of 10 samples or less	< 0.1 mg/l
1 set of EPA Nutrient QC reference samples-conc. 1 and 2, or EPA F/NO_3 QC sample, WS series Conc. 1 and 2	1 per sample set	85% - 115%

*Matrix spike concentrations will be 30% or larger, of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

I. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact ~~Jay Thakkar (312) 886-1972~~ ~~or Chuck Elly (312) 353-9087~~. Sample Management Office

ease return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/013__-0-6/87 Ammonia in water 6/30/87

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling _____ _____

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: 312/~~886-1971~~ 353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II ~~REM~~ REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of ammonia in waters (surface waters, ground waters, drinking waters, etc.) All samples will be unfiltered. Results will be reported as mg/l N.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blanks.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4387

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples: analysis within 28 days and reported 30 days following receipt of samples

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) EPA Method 350.1 (Automated Phenate), or

2) EPA Method 350.3 (Potentiometric, Ion Selective Electrode).

Samples will be stored at 4° C until analysis and validation of results. Sample aliquots will be preserved in the field with sulfuric acid (1 ml/l to pH < 2).

The working concentration range of Method 350.1 Auto Analyzer should be 0.1 to 10 mg/l NH_3-N or lesser concentration. Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample Management Office Check sample pH (wide range pH paper). If pH >2 contact ~~Jay Thakkar, CPMS, CRL~~ for instructions. Dilute and rerun samples with peak heights or concentrations higher than the highest standard. The holding time is not to exceed 28 days from sample collection. All solutions should be made with amonia-free water. For Method 350.3 calibrate the electrometer with standards in order of increasing concentration of ammonia. The pH of the solution after the addition of NAOH must be above 11. Use only the method(s) specified above. Standard curve for Method 350.1 must include at least 5 standards (one of which is zero concentration). Standard curve for Method 350.3 must include at least 4 standards between 0.1 and 10.0 mg/l NH3-N. All standards, blanks, dilution water, and diluted samples must be acidified with 1 ml/l H_2SO_4.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The test procedure used will be clearly identified. Bench records tabulating the order of calibration standards, lab blanks, samples, lab control standards, spikes, duplicate, etc. with resulting peak heights, millivolts, or concentration readouts, will be provided along with copies of worksheets used to calculate ammonia results. If Method 350.3 is used, the standard curve should be provided. A photocopy of the instrument readout i.e. strip charts, printer tapes, etc. must be included. All records analyses and calculation must be legible and sufficient to recalculate all concentrations. Results are to be in mg/-N per liter.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used. See also Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):
Also see Attachment C.

11. Name of sampling/shipping contact: 312- 474- 1313

Phone: YAN YAN MA

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Ammonia	0.1 mg/l-N	Duplicate results must agree to within 10% for concentrations > 1mg/l or to within 0.1mg/l for concentrations <1 mg/l Results will be reported to the nearest 0.05 mg/l and to 2 significant figures for concentrations exceeding 1/mg/l-N.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

GENERAL STATEMENT

II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.

a) For Method 350.1

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± 10% or 0.1 mg/l
Lab Blank	at least 1 per group of 10 or fewer samples	≤0.1 mg/l
Calibration verification	1 per group of 10 samples	90% - 110%
1 set of EPA QC Nutrient reference samples. Conc. 1 & 2	1 per sample set	85% - 115%

b) For Method 350.3

Lab Duplicate	at least 1 per group of 10 or fewer samples	10% or 0.1 mg/l
Lab Blank	at least 1 per group of 10 or fewer samples	≤ 0.1 mg/l
Calibration verification standard	1 per 10 samples and end of set	90% - 110%
1 set of EPA QC Nutrient reference samples. Conc. 1 & 2.	1 per sample set	85% - 115%

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

I. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Sample Management Office ~~Jay Thakkar (312) 886-1972) or Chuck Elly (312) 353-9087~~.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4389

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/015__-0-7/87

Total Kjedahl Nitrogen in Water
July 30, 1987

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Scheduled for Approval

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: (312) ~~886-1971~~ 353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATIO

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for total Kjeldahl nitrogen in waters (surface waters, groundwaters, drinking waters, leachates, etc.). All samples will be unfiltered. Results will be reported as mg/l N.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 Sample - 25 investigative, 3 field duplicates, 3 field blanks

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4391

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: DAILY BY OVERNIGHT CARRIER

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratories shall report results within 30 days after receipt of samples

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) EPA Method 351.2 (Colorimetric, Block Digestor, AA II)

2) EPA Method 351.3 (Colorimetric, Titrimetric, or Potentiometric) (NOTE: For Method 351.3 the micro-Kjeldahl technique is not acceptable.) Samples will be preserved in the field using H2SO4 (1ml/L) to pH<2, samples will be stored at 4°C until analysis and validation of results. Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

For all Methods: Analyze samples within 28 days after collection. Check the sample pH (wide range pH paper). If the pH>2, contact CPMS, CRL for instructions. Use nicotinic acid for the control standard. Use an organic nitrogen compound for the matrix spike. Use only the Methods specified in item 7. Method 351.3 requires distillation separation, prior to all final ammonia measurements.
For Method 351.3: Use only the Colorimetric method for samples containing less than 1 mg N/l.
For Colorimetric Methods (351.2 and 351.3): Use at least five calibration standards (including a zero concentration standard). Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Potentiometric Method (351.3): Use at least four calibration standards. Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Titrimetric Method (351.3): Standardize the titrant each day. Include records of indicator blank.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the test procedure and options used. Provide bench records and all records of calibration, analyses, and calculations for standards, samples, blanks, any titration indicator blanks, duplicates, spikes, controls, etc. Include absorbances, peak heights, responses, concentrations, etc. for each measurement. Include digestion logs showing sample volumes and dilutions for all samples. Identify organic nitrogen compound used for matrix spikes. Records must be legible and sufficient to recalculate all concentrations and QA audit results. Provide photocopies of all instrument readouts (i.e. stripcharts, print-outs, etc). Report results as mg N/l. Identify the compound used for the matrix spike.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "tru" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used. Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

US4392

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
TKN	0.1 mg N/l	Duplicate sample results must agree within 0.1 mg/l for concentrations <1 mg/l and within 10% for concentrations > or = to 1 mg/l
NOTE: These are minimum requirements. Report the actual detection limit used based on allowable methodology options.		

II. QC REQUIREMENTS Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Control standards (Nicotinic Acid)	one per set	70 - 110% recovery
Matrix spike*	one per group of 10 or fewer samples	85 - 115% recovery
Lab duplicate	" "	± (10% or 0.1 mg N/l)
Lab blank	" "	± 0.1 mg N/l
Calibration verification Standard	" " and at the end of the set	90 - 110%
1 set of EPA QC nutrient reference samples conc. 3 and 4.	one per set	85 - 115%

*Matrix spike concentration will be greater than 30% of the sample concentration but will not exceed the highest calibration standard. Matrix spikes will be prepared from an organic nitrogen compound.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact ~~Chuck Elly (312) 353-9087 or Jay Thakkar (312) 886-1972.~~ Sample Management office

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/008__-0-7/87 Fluoride in Water July 30, 1987

.S. Environmental Protection Agency
LP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
IONE: (703)/557-2490 or FTS/557-2490

SAS Number

Scheduled for Approval _____ _____

SPECIAL ANALYTICAL SERVICES
Client Request

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: ~~(312) 886-1971~~ 312-353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATIO

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

General description of analytical service requested:

Analysis of fluoride in water (residential well water, groundwater, surface water, etc.). Samples will be unfiltered. This method should not be used for (1) wastewaters, (2) waters contaminated by industrial fluoride wastes, and (3) waters of large suspended solids content, all of which require Bellack distillation (See Method 340.1) prior to analysis. Do not use this SAS for waters containing more than 3.0 mg/l AL. Results will be reported as mg/l F.

Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blank.

Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4395

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: DAILY BY OVERNIGHT CARRIER

6. Number of days analysis and data required after laboratory receipt of samples:
LABORATORY ~~SHOULD~~ REPORT ANALYSIS
30 DAYS FROM RECEIPT OF SAMPLES

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 340.2 (Potentiometric, Selective Ion Electrode)
   a). Automated Electrode Method, USGS-I-4327-78 is an acceptable alternative.
   b). Auto Analyzer Method 413E, Standard Methods, 16th ed, is an acceptable alternative.

Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Calibration curve for electrode methods must contain at least 4 standards between 0.1 mg/l and the highest concentration standard. Additional standards must be used to define any non-linearity of electrode response between 0.1 and 0.5 mg/l fluoride. For Method 413E, the calibration curve must contain 5 or more points (including a zero concentration standard). Dilute and reanalyze any samples having an electrode or colorimeter response greater than highest standard. Obtain approval of CPMS, CRL prior to use of any other method.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The test procedure used will be clearly identified. Bench records tabulating order of calibration standards, verification and control standards, blanks, samples, matrix spikes, etc. will be provided with resulting voltages, concentration, peak height, or absorbance readouts will be provided with copies of worksheets used to calculate results. A description of the fluoride electrode instrumentation system will be provided A photocopy of instrument readouts ie stripcharts, printer tapes, etc. must be included for all analyses. All records of analysis and calibrations must be legible and sufficient to recalculate all sample concentrations and QA audit results. EPA QC reference samples, or any other reference sample or or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312- 474- 1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Fluoride	0.10 mg/l	Differences in duplicate sample results should be <0.10 mg/l for concentrations <1.0 mg/l and <10% for concentrations >1.0 mg/l. Report concentrations to nearest 0.01 mg/l between 0.10 and 2.0 mg/l and to 2 significant figures >2.0 mg/l.

II. QC REQUIREMENTS - Do not use designed field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	At least one per group of 10 or fewer samples.	85-115% Recovery
Lab Duplicate	(Same as above.)	+or-(10% or 0.10mg/l)
Lab Blank	(Same as above.)	<0.10 mg/l
Calibration Verification Standard 1 set of EPA QC Mineral Reference Samples or 1 set of EPA WS QC F/NO_3 Reference Samples -2 Concentrates.**	(Same as above.)	90-110% Recovery
	1 per sample set	85-115% Recovery

*Matrix Spike concentrations will be greater than 30% of the sample concentration, but spiked sample will not exceed the working range of the standard curve.

** - Critical requirement.

I. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact ~~Jay Thakker (312)886-~~ Sample Management Office 1972 ~~or Chuck Elly (312) 353-0087~~.

Please return this request to the Sample Management Office as soon as possible to expedite ocessing of your request for special analytical services. Should you have any questions need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/007__-0-6/87

Analysis of Silica

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATIO

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

F. General description of analytical service requested: Analysis of silica in surface waters, groundwater, or drinking water using optimum sensitivity, precision, and accuracy of analysis. Samples will be unfiltered and will not be wastewaters. Results are reported as mg/l silica.

G. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blank.

H. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund Remedial

US4399

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

14 days for analysis. Final report and data within 30 days.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): EPA Method 370.1 (0.45-micron filtration followed by colorimetric, manual).

Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records tabulating the order of calibration standards, verification and control standards, samples, blanks, matrix spikes, etc. with resulting peak height, concentration, or absorbance read-outs will be provided with copies of worksheets used to calculate results. A photocopy of instrument readouts, i.e. stripcharts, printer tapes, etc., must be included with all results. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results.

EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: Yan Yan Ma

Phone: (312) 474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Silica	0.10 mg/l	Differences in duplicate sample results are to be < or = to 0.10 mg/l for concentrations <1.0 mg/l and < or = to 10% for concentrations exceeding 1.0 mg/l.
NOTE: These are minimum requirements. Report actual detection limit used based on allowable methodology options.		Report silica concentrations to the nearest 0.01 mg/l between 0.1 and 2.0 mg/l and to 2 significant figures >2.0 mg/l.

II. QC REQUIREMENTS Do not use any designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix spike*	1 per group of 10 or fewer samples	85 - 115% Recovery
Lab duplicate	" "	+ or -(10% or 0.5 mg/l)
Lab blank	" "	< 0.5 mg/l
Calibration verification standard	" "	90 - 110% Recovery
1 set of EPA QC Mineral Reference samples - 2 concentration levels.	1 per sample set	85 - 115% Recovery

* Matrix spike concentrations will be greater than 30% of sample concentration, but spiked sample shall not exceed working range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/011__-0-7/87 Sulfate in Water July 30, 1987

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: (312) ~~886-1971~~ 353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for sulfate in water (surface water, groundwater, drinking water, leachate, etc.). Samples will be unfiltered. Results are reported as mg/l SO_4.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blank.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4403

5. Estimated date(s) of collection: April 1993

6. Estimated date(s) and method of shipment: DAILY BY OVERNIGHT CARRIER

6. Number of days analysis and data required after laboratory receipt of samples:
analysis of samples 28 days following receipt of samples
results of analysis reported 30 days following receipt of samples

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 375.2 (Colorimetric Methylthmol Blue) - 1983 ed.
- Note: This method requires 0.75 mg/l SO_4 in Dilution Water(See Reagent Section 6.8)

2. Method 426C of Standard Methods, 16th ed. (Turbidimetric)
- Note; this last method provides for measurement of sulfate using 2 standard curves- 1 for sulfate concentrations between 0 and 10mg/l, and 1 between 10 and 40 mg/l sulfate.

Samples will be kept at 4°C until validation of results.

Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
Sample holding time is not to exceed 28 days from date of sample collection. Sulfate standards will be prepared daily from stock solution. Samples with absorbances or turbidities greater than that in the highest standard will be diluted and rerun. For Method 426C, 1) the reanalysis solution should contain between 20 and 40 mg/l sulfate, and 2) concentrations must be corrected for background turbidity and color per Section 5d of Method 426C using pH adjusted sample aliquots. Use only the methods specified. Calibration curves must include at least 6 points (including a zero concentration standard) for Method 375.2 and Buffer A of Method 426C.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.
The test procedure used must be clearly identified. Results shall be reported as mg/l SO_4. Bench records tabulating the order of calibration standards, lab control standards, lab blanks, samples, spikes, etc., with resulting absorbances or concentration readouts, will be provided along with copies of worksheets used to calculate results. Background absorbances used for turbidity corrections must be tabulated for each sample aliquot tested. A photocopy of the instrument readout (ie. strip charts, printer tapes, etc.) must be included. All records of analysis must be legible and sufficient to calculate all concentrations and results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source,lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used. Also see Attachment B

10. Other (use additional sheets or attach supplementary information, as needed):
See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA
Phone: 812- 474- 1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Sulfate	5 mg/l	Method 375.2: Differences in duplicate sample results are to be < 5 mg/l for concentrations < 50 mg/l, and < 10% for concentrations > 50 mg/l.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		Method 426 C: Differences in duplicate sample results are to be ≤ 2 mg/l for concentrations ≤ 20 mg/l and ≤ 10% for concentrations > 20 mg/l in aliquot tested.

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85-115%
Lab Duplicate	" "	± (10% or 5 mg/l) for Method 375.2
		± (10% or 2 mg/l) for Method 426C
Lab Blank (0 mg/l SO_4)	" "	< 5 mg/l - Method 375.2
		-2 to +2mg/l-Buffer B of Method 426C or
Lab Blank (10 mg/l SO_4)	" "	8 to 10mg/l - Buffer A of Method 426C
Calibration Verification Standard	1 per group of 10 samples and at end of sample set	90 - 110%
1 Set of EPA QC Mineral Reference Samples	once per sample set	85-115% for each concentration.

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples shall not exceed working range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087~~. Sample Management Office.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4405

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/007__-0-6/87 Analysis of Chloride 6/25/87

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling

X	Regional Transmittal		Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: ~~(312) 886-1971~~ 312- 353- 2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for our request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of chloride in surface waters, groundwater, or drinking water using optimum sensitivity, precision, and accuracy of analysis. Samples will be unfiltered and will not be wastewaters. Results are reported as mg/l Cl.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blank.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4407

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:
Final report and data due within
14 days for analysis. ~~and~~ 30 days from receipt of samples
~~for report of results~~.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA II), 1983 ed.
NOTE: A Region V CRL Auto-Analyzer manifold (0-20 mg/l) is attached for modification of Method 325.2 and to correct errors in Method 325.2.

or

2. ASTM D 512C-81 (Colorimetric, Manual Ferricyanide).

Samples will be kept at 4°C until analysis and validation of results.

Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): Prepare all standards, reagents, blanks, etc. with ASTM Type II reagent water or equivalent, calibration standards will be prepared daily from stock solutions. Use working concentration range or standard curve between 0-20 mg/l or less. Calibration curves must contain at least 5 points (including a zero concentration standard). Dilute and reanalyze any samples with concentrations greater than highest standard. Remove any large amounts of turbidity prior to sample analysis (see Section 7.1 of Method 325.2).
Use only the specified methods. No others are allowed.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records tabulating the order of calibration standards, verification and control standards, samples, blanks, matrix spikes, etc. with resulting peak height, concentration, or absorbance read-outs will be provided with copies of worksheets used to calculate results. A photocopy of instrument readouts, i.e. stripcharts, printer tapes, etc., must be included with all results. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used. Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
chloride	0.5 mg/l	Differences in duplicate sample results are to be < or = to 0.5 mg/l for concentrations < 5 mg/l and < or = to 10% for concentrations exceeding 5 mg/l.
NOTE: These are minimum requirements. Report actual detection limit used based on allowable methodology options.		Report chloride concentrations to the nearest 0.1 mg/l between 0 and 20 mg/l.

II. QC REQUIREMENTS Do not use any designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix spike*	1 per group of 10 or fewer samples	85 - 115% Recovery
Lab duplicate	" "	+ or -(10% or 0.5 mg/l)
Lab blank	" "	< 0.5 mg/l
Calibration verification standard	" "	90 - 110% Recovery
1 set of EPA QC Mineral Reference samples - 2 concentration levels.	1 per sample set	85 - 115% Recovery

* Matrix spike concentrations will be greater than 30% of sample concentration, but spiked sample shall not exceed working range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087~~. Sample Management Office

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4409

5/016__-0-6/87 Total Phosphorus in H_2O 6/29/87

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

Approved For Scheduling

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: (312) ~~886-1971~~ 353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for total phosphorous in waters (surface waters, ground waters, drinking waters, leachate, etc.). Most samples will be unfiltered although certain aliquots can be filtered and preserved at time of collection. Results will be reported as mg/l P.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 samples - 25 investigative, 3 field duplicates, 3 field blanks.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4410

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: DAILY BY OVERNIGHT CARRIER

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days after receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Total Phosphorus EPA Method 365.1 (Automated, Colorimetric, Ascorbic Acid)

Total Phosphorus EPA Method 365.2 (Automated, Colorimetric, Single Reagent)

Total Phosphorus EPA Method 365.4 (Block Digestor)

Samples will be preserved in the field with 1 ml/l H_2SO_4 to pH <2 and stored at 4°C until analysis and validation of results.

Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.) Check sample pH using wide-range pH paper. If the pH>2, contact CPMS, CRL for instructions:

Dilute and redigest samples with absorbances or peak heights higher than the highest standard. All standards, blanks, audits, etc. must be digested. The holding time is not to exceed 28 days from sample collection. Use only the method(s) specified above. The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The test procedure used will be clearly identified. Bench records and all records of analysis and calculations for samples, blanks, duplicates, spikes and all control checks with peak height or response and concentrations will be provided with copies of worksheets. Results will be reported as mg/l P. Any digestion log will be provided showing sample aliquots and concentrations of all samples tested. Records must be legible and sufficient to recalculate all concentrations. A photocopy of the instrument readout i.e. stripcharts, printer tapes, etc. must be included. EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

Also see Attachment B

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312- 474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Total P	0.05 mg/l	Duplicate results must agree to within 10% for concentrations > 0.5 mg/l or within 0.05 mg/l for concentrations < 0.5 mg/l
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± (10% or 0.05 mg/l)
Lab Blank (Also serves as a calibration blank).	at least 1 per group of 10 or fewer samples	<0.05 mg/l
Calibration verification standard	1 per group of 10 samples and end of sample set	90% - 110%
1 set of EPA nutrient QC reference samples conc. 3&4	1 per sample set	85% - 115%

*The matrix spike concentrations will be approximately 30% or larger of sample concentrations, but spiked samples shall not exceed the working range of the standard curve.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087~~ Sample Management Office.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

7018__-0-6/87 COD (Hi- and Lo-levels) 6/26/87

S. Environmental Protection Agency
P Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request Approved for Scheduling

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

RSCC Representative: ~~Dennis Wesolowski~~ Jan Pels

C. Telephone Number: ~~312/886-1971~~ 312-353-2720

Date of Request: March 1993

Site Name: Southeast Rockford Phase II Remedial Investigation

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of chemical oxygen demand (COD) (Hi-level) (50 - 800 mg/l) and COD (Lo-level) (5 - 50 mg/l) in water (surface water, groundwater, leachate, wastewater, drinking water, etc.). Samples will be unfiltered. Any sample with COD values less than 50 mg/l will be determined and reported as COD (Lo-level). Samples with COD values greater than or equal to 50 mg/l will be determined and reported as COD (Hi-level). The COD (Lo-level) method is used for optimum precision and accuracy of measurement of low concentration COD values.

If field specific conductance values are greater than or equal to 5000 umhos/cm, field personnel will indicate this on the SAS Packing Lists or any future RAS/SAS Traffic Report Forms, the field conductance values and the instruction "Check for Possible Chloride Interference in COD Test" for any such samples. If this is the case or if there is other definite knowledge of chlorides exceeding 2000 mg/l, the laboratory will determine the chloride content and inhibit chloride interference pursuant to Section 7.1 of EPA Method 410.3.

NOTE: It is expected that few waters from Region V (<3%), will have chloride concentrations in excess of 2000 mg/l, however, it is the responsibility of field personnel to first identify any samples having a chloride interference for COD so that the COD test can be modified to compensate for any chloride interference.

Results are reported as mg/l COD.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration): 31 aqueous samples

Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.): Superfund Remedial

US4414

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier.

6. Number of days analysis and data required after laboratory receipt of samples:
Report results within 30 days.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 410.1 (Titrimetric, Mid-level) for COD $\geq$ 50 mg/l.
EPA Method 410.2 (Titrimetric, Low-level) for COD $<$ 50 mg/l.
Use Section 7.1 of Method 410.3 if chloride concentration exceeds 2000 mg/l in a sample. If titration blank is necessary for each different amount of mercuric sulfate used for inhibition of chloride interference, SAS Packing Lists will note the samples requiring assessment of chloride interferences. Measurement of chloride will be done using any method of "Standard Methods",16th ed., or "EPA Methods for Chemical Analysis of Water and Wastes", 1983 ed., whenever possible chloride interference is noted.
Samples will be preserved with 1 ml of H_2SO_4 to pH less than 2 and kept at 4°C until sample analysis and validation of results are completed. Holding time is not to exceed 28 days from date of sample collection.
Also see Attachment A

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Check sample pH (wide range pH paper). If pH>2, contact ~~GPMS, CRL~~ Sample Management Office for further instructions.
2. Use a) 50 ml sample aliquots for both methods, b) 0.250 N $K_2Cr_2O_7$ reagent and 0.25 N ferrous ammonium sulfate titrant for Method 410.1, and c) 0.0250 N $K_2Cr_2O_7$ reagent and 0.025 N ferrous ammonium sulfate titrant for Method 410.2.
3. Dilute and reanalyze (by Method 410.1) any samples with COD values > 800 mg/l or titrant volumes < 5.0 ml. Reanalyze samples (by Method 410.1) if initial sample values are > 50 mg/l COD by Method 410.2. Reanalyze samples (by Method 410.2) if initial sample values are < 50 mg/l COD by Method 410.1.
4. Any sample aliquots < 50 mls will be diluted to 50 mls so that the COD reaction mixture will be 50% H_2SO_4/ 50% water by volume.
5. Titration blanks will be determined, at least in duplicate each day of analysis and will not differ more than $\pm$ 0.1 ml titrant for Method 410.1 and $\pm$ 1.0 ml titrant for Method 410.2.
6. Separate sets of QA Audits will be performed for each method, if both methods are used.
7. Use potassium hydrogen phthalate as a matrix spike compound. Use 20 mg/l matrix spike concentration for Method 410.2.
8. Samples will be refluxed for at least 2 hours.
9. Homogenize sample aliquots, as necessary, to obtain sample aliquots of representative suspended solids.
10. Use only the method specified.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records, tabulating titrant standardization, titration volumes for titration or sample blanks (2 or more in number), samples, and QA Audits will be provided for each method used. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results. Records of chloride analysis will be provided for any samples so specified on the RAS/SAS Traffic Report or SAS Packing List. Separate bench records will be provided for any COD determinations of high chloride samples (>2000 mg/l Cl) including weight of mercuric sulfate used, sample titration volume and titration blank volume for each sample type. EPA QC Reference samples, or any other reference samples, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: Yan Yan Ma

Phone: 312-474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
COD (Method 410.1)	50 mg/l	Method 410.1: Differences in sample duplicates are to be < or = to 0.2 ml titrant or ≤ 8 mg/l for concentrations ≤ 80 mg/l and < 10% for COD concentrations exceeding 80 mg/l.
COD (Method 410.2)	5 mg/l	Method 410.2: Differences in sample duplicate results are to be ≤ 1.0 ml titrant or ≤ 4 mg/l for concentrations less than 40 mg/l and are to be ≤ 5 mg/l for concentrations between 40 50 mg/l.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix spike (KHP) Method 410.1* Method 410.2(Use 20 mg/l spike)	at least 1 per group of 10 or fewer samples	85 - 115% Recovery (410.1) 75 - 125% Recovery (410.2)
Lab duplicate	" "	Diff ≤ (8 mg/l or 10%) (410.1)
	" "	Diff ≤ (4 mg/l - 5 mg/l) (410.2)
Titration blank (used for calculation of results)	at least 2 per sample set for each method used	Diff in titrant volumes shall not exceed 0.1 ml for 410.1 and 1.0 ml for 410.2
1 set of EPA QC Demand Reference samples - 2 concentration levels	1 per sample set for each method used	90 - 110% Recovery or ≤ 8 mg/l error for 410.1 and ≤ 5 mg/l error for 410.2 in aliquot tested

* - Matrix spike will be greater than 30% of the sample concentration, but spiked sample shall not exceed 800 mg/l for Method 410.1.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact Sample Management Office ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.~~ Jan Pels (312) 353-2720

Contact Region V RSCC ~~Dennis Wesolowski (312) 886-1971~~ concerning questions on chloride interferences and modifications of COD test.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4417

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/020__-0-6/87 Total Organic Carbon in Water 6/30/87

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request Approved For Scheduling

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: ~~Dennis Wesolowski~~ JAN PELS

C. Telephone Number: ~~(312) 886-1971~~ 312-353-2720

D. Date of Request: March 1993

E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for total organic carbon in water (surface waters, groundwaters, drinking waters, leachate, etc.). Most samples will be unfiltered, although certain aliquots can be filtered and preserved at the time of collection. Results are reported as mg/l C.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 aqueous samples

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

US4419

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier.

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 415.1 (combustion or oxidation).

Samples will be preserved with 1 ml/l H_2SO_4 to pH <2. Samples will be stored at 4°C until analysis and validation of results.

Also see Attachment A.

8. Special technical instruction (if outside protocol requirements) dilute and rerun samples with absorbances higher than the highest standard:

SAMPLE MANAGEMENT OFFICE Check sample pH with (wide range pH paper). If pH >2 contact ~~CPMS, CRL~~ for instructions. The holding time is not to exceed 28 days from sample collection. Homogenize samples if necessary. Qualify results where suspended solids content may affect accuracy. Instruments with syringe injection will utilize 2 injections per measurement. If the 2 injections differ by more than 10% or 2 mg/l, repeat and report the average of 4 injections. Inorganic carbon will be purged from solution or, if determined separately, subtracted from total carbon values. Obtain approval of CPMS, CRL, prior to use of any other method. The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

Test procedures and specific instrument used will be clearly identified. Bench records tabulating order of calibration standards, lab blanks, samples lab control standards, spikes, duplicates etc., with resulting output on concentration readout will be provided along with worksheets used to calculate results. Specify the organic compound used to prepare standards and spikes. A photocopy of the instrument read-out, i.e. stripcharts, printer, tapes, etc. must be included. Results are to be reported in mg/l C. Records of analysis and calculations must be legible and sufficient to re-calculate all concentrations.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.
Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
TOC	2 mg/l	Difference in duplicate results should not exceed ± 10% for concentrations >20 mg/l or 2 mg/l for concentrations less than 20 mg/l.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± (10% or 2.0 mg/l)
Lab Blank	at least 1 per group of 10 or fewer samples	≤ 2.0 mg/l
Calibration verification standard	1 per group of 10 samples and end of set	90% - 110%
1 set of EPA demand QC reference samples (conc. 1 and 2)	1 per sample set	85% - 115%

*The matrix spike concentrations will be approximately 30% of sample concentrations, but spiked samples shall not exceed the working range of the standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Sample Management Office ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087~~.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4421

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/024__-0-6/87 Total Dissolved Solids 6/29/87

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
Phone: (703) 557-2490 or FTS-557-2490

SAS Number

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V
B. Regional Representative: ~~Dennis Wesolowski~~ JAN PELS
C. Telephone Number: (312) ~~886-1971~~ 353-2720
D. Data request: March 1993
E. Site Name: ~~SE~~ SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of total dissolved solids (180°C) in water (surface waters, wastes, groundwaters, drinking water, leachate, etc.) Results are reported as mg/l dissolved solids.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 aqueous samples

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: DAILY BY OVERNIGHT CARRIER

US4423

6. Approximate number of days results required after lab receipt of samples: _____
Laboratory Results reported within 30 days of receipt of Samples

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.1, 1983 ed., or

2. Method 209B, "Standard Methods", 16th ed. Samples will be kept at 4°C until sample analysis and validation of results. Holding time is 7 days from date of sample collection.

Also see Attachment A.

8. Specail technical instructionns (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1) Use standard aliquots of 100ml; however do not use sample aliquots yielding more than 200 mg residue. If residue is greater than 200 mg, repeat the analysis using a smaller sample aliquot. 2) If the pH value is less than 4.0, raise the pH of the aliquot (using NaOH titrant) to between pH 4 and 8 and subtract the weight of sodium added from the weigth of the residue. 3) Residue will be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 the final weight is to be used for calculations. Constant weight is defined as a) less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller, or b) dried overnight (12 hours drying time) with a single weight used for calculations.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the QC reference sample lot numbers used and their true values with 95% confidence intervals. Bench records of tare weights, final weights, additional weights to determine constant weights, volumes filtered, blanks, duplicate samples, and reference samples will be provided with copies of work sheets used to calculate results . Dates and time of 1) determination of tare weights, 2) sample filtration, and 3) determination of residue weights and constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

Also see Attachment B

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4424

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
TDS	20 mg/l	Difference in duplicate sample aliquots shall not exceed 2 mg for residues. Duplicate differences shall not exceed 10% for sample values greater than 200 mg/l.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

II. QUALITY CONTROL REQUIREMENTS Do not use any designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1. 1 set of EPA QC Mineral Reference Samples*- 2 concentration levels.	1 per sample set	85-115% Recovery
2. Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2 mg of residue)
3. Lab Blanks (100 ml of filtered reagent water)	At least 1 per group of 10 or fewer samples	- 20 mg/l to + 20 mg/l

* Alternate reference samples must be approved by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contant ~~Charles T. Elly (312/353-9087) or Jay Thakkar at (312/886-1972).~~ Sample management Office

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

5/025__-[illegible]/87 Total suspended solids in water 6/29/87

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
Phone: (703) 557-2490 or FTS-557-2490

SAS Number

Approved for Scheduling

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V
B. Regional Representative: ~~Dennis Wesolowski~~ JAN PELS
C. Telephone Number: ( ) ~~312/886-1971~~ 312-353-2720
D. Data request: March 1993
E. Site Name: SOUTHEAST ROCKFORD PHASE II REMEDIAL INVESTIGATION

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for total suspended solids (103-105°C) in water (surface waters, groundwater, drinking water, leachate, etc.) Results are reported as mg/l total suspended solids.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

31 aqueous samples

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

SUPERFUND REMEDIAL

4. Estimated date(s) of collection: April 1993

5. Estimated date(s) and method of shipment: Daily by overnight carrier.

US4427

6. Approximate number of days results required after lab receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.2, 1983 ed., (Gravimetric, Dried at 103° - 105° C) using glass fiber filter discs without organic binder such as: Millipore AP-40, Reeve Angel 934-AH, Gelman A/E, or equivalent. Use only membrane filter apparatus with 47 mm diameter glass fiber filter and a coarse (40-60 micron) fritted disc filter support. The filter and support specifications are mandatory. Samples will be held at 4°C until sample analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

Also see Attachment A.

8. Specail technical instructionns (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Sample aliquot volumes are selected on the basis of the following factors. a) During initial sample filtratrion, filtration rate should not drop rapidly, or require more than 5 minutes of filtratrion time. (Increase the filter area or decrease the sample volume as needed for sample reanalysis), b) The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200ml in volume, and c) Sample aliquots should not exceed 200ml in volume. 2. Duplicate sample aliquots will be filtered with 2 or more intervening samples. 3. Final residues are to be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 (The final weight is to be used for calculations), or dried overnight (12 hours of drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller. 4. Use only the method specified above in items 7 and 8.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify EPA QC reference sample lot numbers used and their true values and 95% confidence intervals. Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples (all in the order filtered) will be provided along with copies of worksheets used to calculate results. Dates and time of a) filtration of initial 100ml volume, b) determination of tare weights, c) sample filtration, and d) determination of constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

Also see Attachment B.

10. Other (use additional sheets or attach supplementary information, as needed):

See Attachment C.

11. Name of sampling/shipping contact: YAN YAN MA

Phone: 312-474-1313

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

US4428

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Suspended Solids	2-3 mg/l for 200 ml	Difference in duplicate results shall not exceed 0.5 mg for duplicate aliquots filtered.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.	sample aliquot	

II. QUALITY CONTROL REQUIREMENTS Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1) Lab Duplicates (See item 8.3 on Page 2)	1 per group of 10 or fewer samples	less than 0.5 mg for residue ≤ 5 less than 10% for sample. resid ≥ 5 mg
2) Lab Blanks (200 ml aliquots)	1 per group or 10 or fewer samples	-0.5 to +0.5 mg
3) 1 set of 2 EPA QC Residue Reference Samples-2 concentration levels	1 per sample set	< 5 mg/l error for concentrations < to 50 mg/l or < or = to 10% for nominal concentrations > than 50 mg/l

* Alternate reference samples must be approbed by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and reanalyze samples.

Contact ~~Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087~~. SAMPLE MANAGEMENT OFFICE

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virgina 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: V/CDM

B. RSCC Representative: Jan Pels

C. Telephone Number: 353-2720

D. Date of Request: April 1993

E. Site Name: Southeast Rockford Groundwater Contamination

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of ambient air samples for volatile organics, using tenax tubes.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

24 samples - 20 investigative, 2 field blanks and 2 field duplicates for low-concentration VOCs in air.

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):
Remedial Investigation at Superfund Site

10504.13

US4431

4. Estimated date(s) of collections: April 1993

5. Estimated date(s) and method of shipment: Overnite Air

6. Number of days analysis and data required after laboratory receipt of samples:

30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program:

The method of analysis, performance evaluation procedure, and calibration protocol must follow "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air; Method TO1", attached in Appendix. Analysis must follow the method described in Section 11.2.4 and 11.3

Performance evaluation procedure follows section 14.4.5

Calibration procedures should follow Section 11.2 . Also see Attachment A.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1) Continuing calibration performance check with standard listed in Table 3.

2) The laboratory performing analysis will prepare Tenax tubes according to section 9 of the Compenduim of Methods

3) One cartridge per batch will be checked for contamination as per section 14.2 in

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion. See Attachment B.

1) Report all raw data (TIC's and MS for VOA HSL compounds) and raw QA/QC data (quantitation reports) for all samples, blanks, duplicates and matrix spikes.

2) Report calibration method and response factor for calibration standard (include bench sheet).

3) Report calculated system detection limits.

4) Report up to 10 tentatively identified compounds for each tube analysed using the 3 best library searches. See Attached Sheet for 5 and 6.

10. Other (use additional sheets or attach supplementary information, ad needed)
See attached copy of "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air; method TO1 and copies of selected portions from this document.
See Attachment C.

11) Name of sampling/shipping contact: Yan Yan Ma

10504.13 Phone: (312) 474-1313

addition to #7.

Direct injection calibration procedure is not acceptable, system must be calibrated by analysis of spiked Tenax catridges as described in 11.2.4

9. cont...

5) Report results in nanograms of compound(s) per total mass of Tenax in tube.

6) Report data from tuning and mass standardization.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (+% or Conc.)
See list of Compounds of Interest in Table 3		± 20%

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)
a) Tenax Cartridge Check	Each batch	Field blank: 10ng/g of Tenax lab blank; ½min. conc. of compound interest
b) field blank	1 every 10 samples	≤25% of sample amount
c) Parallel Samples (Reproducibility)	1 every 10 samples	±25% agreement
d) Chromatographic Efficiency	Before & After Analysis	per sec. 14.4.2 of Tol
e) System Detection Limit	Each Sample	20 ng
f) Matrix Spike	1 per 10 Samples	80-100% recovery
	100 ng of starred compounds in table 3	

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

10504.13

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TABLE 3

METHOD DETECTION LIMITS FOR COMPOUNDS OF INTEREST

Volatiles	CAS Number	ng of compound/g of Tenax
1. 1,1-Dichloroethane	75-35-3	20
2. trans-1,2-Dichloroethene	156-60-5	20
3. 1,2-Dichloroethane	107-06-2	20
4. 2-Butanone(MEK)	78-93-3	20
5. 1,1,1-Trichloroethane	71-55-6	10
6. Bromodichloromethane	75-27-4	10
7. 1,1,2,2-Tetracchloroethane	79-34-5	10
8. 1,2-Dichloropropane	78-87-5	20
9. trans-1,3-Dichloropropene	10061-01-5	20
10. Trichloroethene	79-01-6	10
11. Dibromochloromethane	124-48-1	10
12. 1,1,2-Trichloroethane	79-00-5	10
13. Benzene	71-43-2	10
14. cis-1,3-Dichloropropene	10061-01-5	10
15. 2-Chloroethyl Vinyl Ether	110-75-8	20
16. Bromoform	75-25-2	10
17. 2-Hexanone	591-78-6	20
18. 4-Methyl-2-pentanone	108-10-1	20
19. Tetrachloroethene	127-18-4	10
20. Toluene	108-88-3	20
21. Chlorobenzene	108-90-7	20
22. Ethyl Benzene	100-41-4	20
23. Styrene	100-42-5	20
24. Total Xylenes		10

* These compounds and detection limits are mandatory performance standards. If the laboratory is not able to meet these required detection limits, it should be stated.

ATTACHMENT A

The laboratory data rejection and non-payment will be recommended if methods other than those specified in this document are used.

ATTACHMENT B

All original raw data, forms, calculation work sheets, instrument readouts, and preparation forms should be submitted with each data set. If originals were submitted in another data package, photocopies may be submitted with a record of the location of the originals.

ATTACHMENT C

All original tags, COC, SAS packing lists, airbills, and other original forms or copies of receiving log book pages pertaining to this SAS shall be submitted to the Region within the time frame.

COMPENDIUM OF METHODS FOR THE DETERMINATION OF TOXIC ORGANIC COMPOUNDS IN AMBIENT AIR - METHOD TO1

METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING TENAX® ADSORPTION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1. Scope

1.1 The document describes a generalized protocol for collection and determination of certain volatile organic compounds which can be captured on Tenax® GC (poly(2,6-Diphenyl phenylene oxide)) and determined by thermal desorption GC/MS techniques. Specific approaches using these techniques are described in the literature (1-3).

1.2 This protocol is designed to allow some flexibility in order to accommodate procedures currently in use. However, such flexibility also results in placement of considerable responsibility with the user to document that such procedures give acceptable results (i.e. documentation of method performance within each laboratory situation is required). Types of documentation required are described elsewhere in this method.

1.3 Compounds which can be determined by this method are nonpolar organics having boiling points in the range of approximately 80° - 200°C. However, not all compounds falling into this category can be determined. Table 1 gives a listing of compounds for which the method has been used. Other compounds may yield satisfactory results but validation by the individual user is required.

2. Applicable Documents

2.1 ASTM Standards:

D1356 Definitions of Terms Related to Atmospheric Sampling and Analysis.

E355 Recommended Practice for Gas Chromatography Terms and Relationships.

2.3 Other documents:

Existing procedures (1-3).
U.S. EPA Technical Assistance Document (4).

3. Summary of Protocol

3.1 Ambient air is drawn through a cartridge containing ~1-2 grams of Tenax and certain volatile organic compounds are trapped on the resin while highly volatile organic compounds and most inorganic atmospheric constituents pass through the cartridge. The cartridge is then transferred to the laboratory and analyzed.

3.2 For analysis the cartridge is placed in a heated chamber and purged with an inert gas. The inert gas transfers the volatile organic compounds from the cartridge onto a cold trap and subsequently onto the front of the GC column which is held at low temperature (e.g. - 70°C). The GC column temperature is then increased (temperature programmed) and the components eluting from the column are identified and quantified by mass spectrometry. Component identification is normally accomplished, using a library search routine, on the basis of the GC retention time and mass spectral characteristics. Less sophistacated detectors (e.g. electron capture or flame ionization) may be used for certain applications but their suitability for a given application must be verified by the user.

3.3 Due to the complexity of ambient air samples only high resolution (i.e. capillary) GC techniques are considered to be acceptable in this protocol.

4. Significance

4.1 Volatile organic compounds are emitted into the atmosphere from a variety of sources including industrial and commercial facilities, hazardous waste storage facilities, etc. Many of these compounds are toxic; hence knowledge of the levels of

such materials in the ambient atmosphere is required in order to determine human health impacts.

4.2 Conventional air monitoring methods (e.g. for workspace monitoring) have relied on carbon adsorption approaches with subsequent solvent desorption. Such techniques allow subsequent injection of only a small portion, typically 1-5% of the sample onto the GC system. However, typical ambient air concentrations of these compounds require a more sensitive approach. The thermal desorption process, wherein the entire sample is introduced into the analytical (GC/MS) system fulfills this need for enhanced sensitivity.

5. Definitions

Definitions used in this document and any user prepared SOPs should be consistent with ASTM D1356(6). All abbreviations and symbols are defined with this document at the point of use.

6. INTERFERENCES

6.1 Only compounds having a similar mass spectrum and GC retention time compared to the compound of interest will interfere in the method. The most commonly encountered interferences are structural isomers.

6.2 Contamination of the Tenax cartridge with the compound(s) of interest is a commonly encountered problem in the method. The user must be extremely careful in the preparation, storage, and handling of the cartridges throughout the entire sampling and analysis process to minimize this problem.

7. Apparatus

7.1 Gas Chromatograph/Mass Spectrometry system - should be capable of subambient temperature programming. Unit mass resolution or better up to 800 amu. Capable of scanning 30-440 amu region every 0.5-1 second. Equipped with data system for instrument control as well as data acquisition, processing and storage.

7.2 Thermal Desorption Unit - Designed to accommodate Tenax cartridges in use. See Figure 2a or b.

7.3 Sampling System - Capable of accurately and precisely drawing an air flow of 10-500 ml/minute through the Tenax cartridge. (See Figure 3a or b.)

7.4 Vacuum oven - connected to water aspirator vacuum supply.

7.5 Stopwatch

7.6 Pyrex disks - for drying Tenax.

7.7 Glass jar - Capped with Teflon-lined screw cap. For storage of purified Tenax.

7.8 Powder funnel - for delivery of Tenax into cartridges.

7.9 Culture tubes - to hold individual glass Tenax cartridges.

7.10 Friction top can (paint can) - to hold clean Tenax cartridges.

7.11 Filter holder - stainless steel or aluminum (to accommodate 1 inch diameter filter). Other sizes may be used if desired. (optional)

7.12 Thermometer - to record ambient temperature.

7.13 Barometer (optional).

7.14 Dilution bottle - Two-liter with septum cap for standards preparation.

7.15 Teflon stirbar - 1 inch long.

7.16 Gas-tight glass syringes with stainless steel needles - 10-500 µl for standard injection onto GC/MS system.

7.17 Liquid microliter syringes - 5.50 µL for injecting neat liquid standards into dilution bottle.

7.18 Oven - 60 $\pm$ 5°C for equilibrating dilution flasks.

7.19 Magnetic stirrer.

7.20 Heating mantel.

7.21 Variac

7.22 Soxhlet extraction apparatus and glass thimbles - for purifying Tenax.

7.23 Infrared lamp - for drying Tenax.

7.24 GC column - SE-30 or alternative coating, glass capillary or fused silica.

7.25 Psychrometer - to determine ambient relative humidity. (optional).

8. Reagents and Materials

8.1 Empty Tenax cartridges - glass or stainless steel (See Figure 1a or b).
8.2 Tenax 60/80 mesh (2,6-diphenylphenylene oxide polymer).
8.3 Glasswool - silanized.
8.4 Acetone - Pesticide quality or equivalent.
8.5 Methanol - Pesticide quality, or equivalent.
8.6 Pentane - Pesticide quality or equivalent.
8.7 Helium - Ultra pure, compressed gas. (99.9999%)
8.8 Nitrogen - Ultra pure, compressed gas. (99.9999%)
8.9 Liquid nitrogen.
8.10 Polyester gloves - for handling glass Tenax cartridges.
8.11 Glass Fiber Filter - one inch diameter, to fit in filter holder. (optional)
8.12 Perfluorotributylamine (FC-43).
8.13 Chemical Standards - Neat compounds of interest. Highest purity available.
8.14 Granular activated charcoal - for preventing contamination of Tenax cartridges during storage.

9. Cartridge Construction and Preparation

9.1 Cartridge Design

9.1.1 Several cartridge designs have been reported in the literature (1-3). The most common (1) is shown in Figure 1a. This design minimizes contact of the sample with metal surfaces, which can lead to decomposition in certain cases. However, a disadvantage of this design is the need to rigorously avoid contamination of the <u>outside</u> portion of the cartridge since the entire surface is subjected to the purge gas stream during the desorption porcess.

Clean polyester gloves must be worn at all times when handling such cartridges and exposure of the open cartridge to ambient air must be minimized.

9.1.2 A second common type of design (3) is shown in Figure 1b. While this design uses a metal (stainless steel) construction, it eliminates the need to avoid direct contact with the exterior surface since only the interior of the cartridge is purged.

9.1.3 The thermal desorption module and sampling system must be selected to be compatible with the particular cartridge design chosen. Typical module designs are shown in Figures 2a and b. These designs are suitable for the cartridge designs shown in Figures 1a and 1b, respectively.

9.2 Tenax Purification

9.2.1 Prior to use the Tenax resin is subjected to a series of solvent extraction and thermal treatment steps. The operation should be conducted in an area where levels of volatile organic compounds (other than the extraction solvents used) are minimized.

9.2.2 All glassware used in Tenax purification as well as cartridge materials should be thoroughly cleaned by water rinsing followed by an acetone rinse and dried in an oven at 250°C.

9.2.3 Bulk Tenax is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in the soxhlet extraction apparatus and extracted sequentially with methanol and then pentane for 16-24 hours (each solvent) at approximately 6 cycles/hour. Glasswool for cartidge preparation should be cleaned in the same manner as Tenax.

9.2.4 The extracted Tenax is immediately placed in an open glass dish and heated under an infrared lamp for two

hours in a hood. Care must be exercised to avoid over heating of the Tenax by the infrared lamp. The Tenax is then placed in a vacuum oven (evacuated using a water aspirator) without heating for one hour. An inert gas (helium or nitrogen) purge of 2-3 ml/minute is used to aid in the removal of solvent vapors. The oven temperature is then increased to 110°C, maintaining inert gas flow and held for one hour. The oven temperature control is then shut off and the oven is allowed to cool to room temperature. Prior to opening the oven, the oven is slightly pressurized with nitrogen to prevent contamination with ambient air. The Tenax is removed from the oven and sieved through a 40/60 mesh sieve (acetone rinsed and oven dried) into a clean glass vessel. If the Tenax is not to be used immediately for cartridge preparation it should be stored in a clean glass jar having a Teflon-lined screw cap and placed in a desiccator.

9.3 Cartridge Preparation and Pretreatment

9.3.1 All cartridge materials are pre-cleaned as described in Section 9.2.2. If the glass cartridge design shown in Figure 1a is employed all handling should be conducted wearing polyester gloves.

9.3.2 The cartridge is packed by placing a 0.5-1cm glass-wool plug in the base of the cartridge and then filling the cartridge to within approximately 1 cm of the top. A 0.5-1cm glasswool plug is placed in the top of the cartridge.

9.3.3 The cartridges are then thermally conditioned by heating for four hours at 270°C under an inert gas (helium) purge (100 - 200 ml/min).

9.3.4 After the four hour heating period the cartridges are allowed to cool. Cartridges of the type shown in Figure 1a are immediately placed (without cooling) in clean culture tubes having Teflon-lined screw caps with a glasswool cushion at both the top and the bottom. Each tube should be shaken to ensure that the cartridge is held firmly in place. Cartridges of the type shown in Figure 1b are allowed to cool to room temperature under inert gas purge and are then closed with stainless steel plugs.

9.3.5 The cartridges are labeled and placed in a tightly sealed metal can (e.g. paint can or similar friction top container). For cartridges of the type shown in Figure 1a the culture tube, not the cartridge,is labeled.

9.3.6 Cartridges should be used for sampling within 2 weeks after preparation and analyzed within two weeks after sampling. If possible the cartridges should be stored at -20°C in a clean freezer (i.e. no solvent extracts or other sources of volatile organics contained in the freezer).

10. Sampling

10.1 Flow rate and Total Volume Selection

10.1.1 Each compound has a characteristic retention volume (liters of air per gram of adsorbent) which must not be exceeded. Since the retention volume is a function of temperature, and possibly other sampling variables, one must include an adequate margin of safety to ensure good collection efficiency. Some considerations and guidance in this regard are provided in a recent report (5). Approximate breakthrough volumes at 38°C (100°F) in liters/gram of Tenax are provided in Table 1. These retention volume data are supplied only as rough guidance and are subject to considerable variability, depending on cartridge design as well as sampling parameters and atmospheric conditions.

10.1.2 To calculate the maximum total volume of air which can be sampled use the following equation:

$$V_{MAX} = \frac{V_b \times W}{1.5}$$

where

V_{MAX} is the calculated maximum total volume in liters.

V_b is the breakthrough volume for the least retained compound of interest (Table 1) in liters per gram of Tenax.

W is the weight of Tenax in the cartridge, in grams.

1.5 is a dimensionless safety factor to allow for variability in atmospheric conditions. This factor is appropriate for temperatures in the range of 25-30°C. If higher temperatures are encountered the factor should be increased (i.e. maximum total volume decreased).

10.1.3 To calculate maximum flow rate use the following equation:

$$Q_{MAX} = \frac{V_{MAX}}{t} \times 1000$$

where

Q_{MAX} is the calculated maximum flow rate in milliliters per minute.

t is the desired sampling time in minutes. Times greater than 24 hours (1440 minutes) generally are unsuitable because the flow rate required is too low to be accurately maintained.

10.1.4 The maximum flow rate Q_{MAX} should yield a linear flow velocity of 50-500 cm/minute. Calculate the linear velocity corresponding to the maximum flow rate using the following equation:

$$B = \frac{Q_{MAX}}{\pi r^2}$$

where

B is the calculated linear flow velocity in centimeters per minute.

r is the internal radius of the cartridge in centimeters.

If B is greater than 500 centimeters per minute either the total sample volume (V_{MAX}) should be reduced or the sample flow rate (Q_{MAX}) should be reduced by increasing the collection time. If B is less than 50 centimeters per minute the sampling rate (Q_{MAX}) should be increased by reducing the sampling time. The total sample value (V_{MAX}) cannot be increased due to component breakthrough.

10.1.4 The flow rate calculated as described above defines the maximum flow rate allowed. In general, one should collect additional samples in parallel, for the same time period but at lower flow rates. This practice yields a measure of quality control and is further discussed in the literature (5). In general, flow rates 2 to 4 fold lower than the maximum flow rate should be employed for the parallel samples. In all cases a constant flow rate should be achieved for each cartridge since accurate integration of the analyte concentration requires that the flow be constant over the sampling period.

10.2 Sample Collection

10.2.1 Collection of an accurately known volume of air is critical to the accuracy of the results. For this reason the use of mass flow controllers, rather than conventional needle valves or orifices is highly recommended, especially at low flow velocities (e.g. less than 100 milliliters/minute). Figure 3a illustrates a sampling system utilizing mass flow controllers. This system readily allows for collection of parallel samples. Figures 3b shows a commercially available system based on

10.2.2 Prior to sample collection insure that the sampling flow rate has been calibrated over a range including the rate to be used for sampling, with a "dummy" Tenax cartridge in place. Generally calibration is accomplished using a soap bubble flow meter or calibrated wet test meter. The flow calibration device is connected to the flow exit, assuming the entire flow system is sealed. ASTM Method D3686 describes an appropriate calibration scheme, not requiring a sealed flow system downstream of the pump.

10.2.3 The flow rate should be checked before and after each sample collection. If the sampling interval exceeds four hours the flow rate should be checked at an intermediate point during sampling as well. In general, a rotameter should be included, as showed in Figure 3b, to allow observation of the sampling flow rate without disrupting the sampling process.

10.2.4 To collect an air sample the cartridges are removed from the sealed container just prior to initiation of the collection process. If glass cartridges (Figure 1a) are employed they must be handled only with polyester gloves and should not contact any other surfaces.

10.2.5 A particulate filter and holder are placed on the inlet to the cartridges and the exit end of the cartridge is connected to the sampling apparatus. In many sampling situations the use of a filter is not necessary if only the total concentration of a component is desired. Glass cartridges of the type shown in Figure 1a are connected using teflon ferrules and Swagelok (stainless steel or teflon) fittings. Start the pump and record the following parameters on an appropriate data sheet (Figure 4): data, sampling location, time, ambient temperature, barometric

pressure, relative humidity, dry gas meter reading (if applicable) flow rate, rotameter reading (if applicable), cartridge number and dry gas meter serial number.

10.2.6 Allow the sampler to operate for the desired time, periodically recording the variables listed above. Check flow rate at the midpoint of the sampling interval if longer than four hours.
At the end of the sampling period record the parameters listed in 10.2.5 and check the flow rate and record the value. If the flows at the beginning and end of the sampling period differ by more than 10% the cartridge should be marked as suspect.

10.2.7 Remove the cartridges (one at a time) and place in the original container (use gloves for glass cartridges). Seal the cartridges or culture tubes in the friction-top can containing a layer of charcoal and package for immediate shipment to the laboratory for analysis. Store cartridges at reduced temperature (e.g. - 20°C) before analysis if possible to maximize storage stability.

10.2.8 Calculate and record the average sample rate for each cartridge according to the following equation:

$$Q_A = \frac{Q_1 + Q_2 + \ldots Q_N}{N}$$

where

Q_A = Average flow rate in ml/minute.
$Q_1, Q_2, \ldots . Q_N$ = Flow rates determined at beginning, end, and immediate points during sampling.

N = Number of points averaged.

10.2.9 Calculate and record the total volumetric flow for each cartridge using the following equation:

$$V_m = \frac{T \times Q_A}{1000}$$

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where

V_m = Total volume sampled in liters at measured temperature and pressure.
T_2 = Stop time.
T_1 = Start time.
T = Sampling time = $T_2 - T_1$, minutes

10.2.10 The total volume (V_s) at standard conditions, 25°C and 760 mmHg, is calculated from the following equation:

$$V_s = V_m \times \frac{P_A}{760} \times \frac{298}{273 + t_A}$$

where

P_A = Average barometric pressure, mmHg
t_A = Average ambient temperature, °C.

11. GC/MS Analysis

11.1 Instrument Set-up

11.1.1 Considerable variation from one laboratory to another is expected in terms of instrument configuration. Therefore each laboratory must be responsible for verifying that their particular system yields satisfactory results. Section 14 discusses specific performance criteria which should be met.

11.1.2 A block diagram of the typical GC/MS system required for analysis of Tenax cartridges is depicted in Figure 5. The operation of such devices is described in 11.2.4. The thermal desorption module must be designed to accommodate the particular cartridge configuration. Exposure of the sample to metal surfaces should be minimized and only stainless steel, or nickel metal surfaces should be employed.

The volume of tubing and fittings leading from the cartridge to the GC column must be minimized and all areas must be well-swept by helium carrier gas.

11.1.3 The GC column inlet should be capable of being cooled to -70°C and subsequently increased rapidly to approximately 30°C. This can be most readily accomplished using a GC equipped with subambient cooling capability (liquid nitrogen) although other approaches such as manually cooling the inlet of the column in liquid nitrogen may be acceptable.

11.1.4 The specific GC column and temperature program employed will be dependent on the specific compounds of interest. Appropriate conditions are described in the literature (1-3). In general a nonpolar stationary phase (e.g. SE-30, OV-1) temperature programmed from 30°C to 200°C at 8°/minute will be suitable. Fused silica bonded phase columns are preferable to glass columns since they are more rugged and can be inserted directly into the MS ion source, thereby eliminating the need for a GC/MS transfer line.

11.1.5 Capillary column dimensions of 0.3 mm ID and 50 meters long are generally appropriate although shorter lengths may be sufficient in many cases.

11.1.6 Prior to instrument calibration or sample analysis the GC/MS system is assembled as shown in Figure 5. Helium purge flows (through the cartridge) and carrier flow are set at approximately 10 ml/minute and 1-2 ml/minute respectively. If applicable, the injector sweep flow is set at 2-4 ml/minute.

11.1.7 Once the column and other system components are assembled and the various flows established the column temperature is increased to 250°C for approximately four hours (or overnight if desired) to condition the column.

11.1.8 The MS and data system are set according to the manufacturer's instructions. Electron impact ionization (70eV) and an electron multiplier gain of approximately 5×10^4 should be employed. Once the entire GC/MS system has been setup the system is calibrated as described in Section 11.2. The user should prepare a detailed standard operating procedure (SOP) describing this process for the particular instrument being used.

11.2 Instrument Calibration

11.2.1 Tuning and mass standarization of the MS system is performed according to manufacturer's instructions and relevant information from the user prepared SOP. Perfluorotributylamine should generally be employed for this purpose. The material is introduced directly into the ion source through a molecular leak. The instrumental parameters (e.g. lens voltages, resolution, etc.) should be adjusted to give the relative ion abundances shown in Table 2 as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source may require cleaning according to manufacturer's instructions. In the event that the user's instrument cannot achieve these relative ion abundances, but is otherwise operating properly, the user may adopt another set of relative abundances as performance criteria.

However, these alternate values must be repeatable on a day-to-day basis.

11.2.2 After the mass standarization and tuning process has been completed and the appropriate values entered into the data system the user should then calibrate the entire system by introducing known quantities of the standard components of interest into the system. Three alternate procedures may be employed for the calibration process including 1) direct syringe injection of dilute vapor phase standards, prepared in a dilution bottle, onto the GC column, 2) Injection of dilute vapor phase standards into a carrier gas stream directed through the Tenax cartridge, and 3) introduction of permeation or diffusion tube standards onto a Tenax cartridge. The standards preparation procedures for each of these approaches are described in Section 13. The following paragraphs describe the instrument calibration process for each of these approaches.

11.2.3 If the instrument is to be calibrated by direct injection of a gaseous standard, a standard is prepared in a dilution bottle as described in Section 13.1. The GC column is cooled to -70°C (or, alternately, a portion of the column inlet is manually cooled with liquid nitrogen). The MS and data system is set up for acquisition as described in the relevant user SOP. The ionization filament should be turned off during the initial 2-3 minutes of the run to allow oxygen and other highly volatile components to elute. An appropriate volume (less than 1 ml) of the gaseous standard is injected onto the GC system using an accurately calibrated gas tight syringe.

The system clock is started and the column is maintained at -70°C (or liquid nitrogen inlet cooling) for 2 minutes. The column temperature is rapidly increased to the desired initial temperature (e.g. 30°C). The temperature program is started at a consistent time (e.g. four minutes) after injection. Simultaneously the ionization filament is turned on and data acquisition is initiated. After the last component of interest has eluted acquisiton is terminated and the data is processed as described in Section 11.2.5. The standard injection process is repeated using different standard volumes as desired.

11.2.4 If the system is to be calibrated by analysis of spiked Tenax cartridges a set of cartridges is prepared as described in Sections 13.2 or 13.3. Prior to analysis the cartridges are stored as described in Section 9.3. If glass cartridges (Figure 1a) are employed care must be taken to avoid direct contact, as described earlier. The GC column is cooled to -70°C, the collection loop is immersed in liquid nitrogen and the desorption module is maintained at 250°C. The inlet valve is placed in the desorb mode and the standard cartridge is placed in the desorption module, making certain that no leakage of purge gas occurs. The cartridge is purged for 10 minutes and then the inlet valve is placed in the inject mode and the liquid nitrogen source removed from the collection trap. The GC column is maintained at -70°C for two minutes and subsequent steps are as described in 11.2.3. After the process is complete the cartridge is removed from the desorption module and stored for subsequent use as described in Section 9.3.

11.2.5 Data processing for instrument calibration involves determining retention times, and integrated characteristic ion intensities for each of the compounds of interest. In addition, for at least one chromatographic run, the individual mass spectra should be inspected and compared to reference spectra to ensure proper instrumental performance. Since the steps involved in data processing are highly instrument specific, the user should prepare a SOP describing the process for individual use. Overall performance criteria for instrument calibration are provided in Section 14. If these criteria are not achieved the user should refine the instrumental parameters and/or operating procedures to meet these criteria.

11.3 Sample Analysis

11.3.1 The sample analysis process is identical to that described in Section 11.2.4 for the analysis of standard Tenax cartridges.

11.3.2 Data processing for sample data generally involves 1) qualitatively determining the presence or absence of each component of interest on the basis of a set of characteristic ions and the retention time using a reverse-search software routine, 2) quantification of each identified component by integrating the intensity of a characteristic ion and comparing the value to that of the calibration standard, and 3) tentative identification of other components observed using a forward (library) search software routine. As for other user specific processes, a SOP should be prepared describing the specific operations for each individual laboratory.

12. Calculations

12.1 Calibration Response Factors

12.1.1 Data from calibration standards is used to calculate a response factor for each component of interest. Ideally the process involves analysis of at least three calibration levels of each component during a given day and determination of the response factor (area/nanogram injected) from the linear least squares fit of a plot of nanograms injected versus area (for the characteristic ion). In general quantities of component greater than 1000 nanograms should not be injected because of column overloading and/or MS response nonlinearity.

12.1.2 In practice the daily routine may not always allow analysis of three such calibration standards. In this situation calibration data from consecutive days may be pooled to yield a response factor, provided that analysis of replicate standards of the same concentration are shown to agree within 20% on the consecutive days. One standard concentration, near the midpoint of the analytical range of interest, should be chosen for injection every day to determine day-to-day response reproducibility.

12.1.3 If substantial nonlinearity is present in the calibration curve a nonlinear least squares fit (e.g. quadratic) should be employed. This process involves fitting the data to the following equation:

$$Y = A + BX + CX^2$$

where

Y = peak area
X = quantity of component, nanograms
A,B, and C are coefficients in the equation

12.2 Analyte Concentrations

12.2.1 Analyte quantities on a sample cartridge are calculated from the following equation:

$$Y_A = A + BX_A + CX_A$$

where

Y_A is the area of the analyte characteristic ion for the sample cartridge.

X_A is the calculated quantity of analyte on the sample cartridge, in nanograms.

A,B, and C are the coefficients calculated from the calibration curve described in Section 12.1.3.

12.2.2 If instrumental response is essentially linear over the concentration range of interest a linear equation (C=0 in the equation above) can be employed.

12.2.3 Concentration of analyte in the original air sample is calculated from the following equation:

$$C_A = \frac{X_A}{V_S}$$

where

C_A is the calculated concentration of analyte in nanograms per liter.

V_S and X_A are as previously defined in Section 10.2.10 and 12.2.1, respectively.

13. Standard Preparation

13.1 Direct Injection

13.1.1 This process involves preparation of a dilution bottle containing the desired concentrations of compounds of interest for direct injection onto the GC/MS system.

13.1.2 Fifteen three-millimeter diameter glass beads and a one-inch Teflon stirbar are placed in a clean two-liter glass septum capped bottle and the exact volume is determined by weighing the bottle before and after filling with deionized water. The bottle is then rinsed with acetone and dried at 200°C.

13.1.3 The amount of each standard to be injected into the vessel is calculated from the desired injection quantity and volume using the following equation:

$$W_T = \frac{W_I}{V_I} \times V_B$$

where

W_T is the total quantity of analyte to be injected into the bottle in milligrams

W_I is the desired weight of analyte to be injected onto the GC/MS system or spiked cartridge in nanograms

V_I is the desired GC/MS or cartridge injection volume (should not exceed 500) in microliters.

V_B is total volume of dilution bottle determined in 13.1.1, in liters.

13.1.4 The volume of the neat standard to be injected into the dilution bottle is determined using the following equation:

$$V_T = \frac{W_T}{d}$$

where

V_T is the total volume of neat liquid to be injected in microliters.

d is the density of the neat standard in grams per milliliter.

13.1.6 The bottle is placed in a 60°C oven for at least 30 minutes prior to removal of a vapor phase standard.

13.1.7 To withdraw a standard for GC/MS injection the bottle is removed from the oven and stirred for 10-15 seconds. A suitable gas-tight microber syring warmed to 60°C, is inserted through the septum cap and pumped three times slowly. The appropriate volume of sample (approximately 25% larger than the desired injection volume) is drawn into the syringe and the volume is adjusted to the exact value desired and then immediately injected over a 5-10 seconds period onto the GC/MS system as described in Section 11.2.3.

13.2 Preparation of Spiked Cartridges by Vapor Phase Injection

13.2.1 This process involves preparation of a dilution bottle containing the desired concentrations of the compound(s) of interest as described in 13.1 and injecting the desired volume of vapor into a flowing inert gas stream directed through a clean Tenax cartridge.

13.2.2 A helium purge system is assembled wherein the helium flow 20-30 mL/minute is passed through a stainless steel Tee fitted with a septum injector. The clean Tenax cartridge is connected downstream of the tee using appropriate Swagelok fittings. Once the cartridge is placed in the flowing gas stream the appropriate volume vapor standard, in the dilution bottle, is injected through the septum as described in 13.1.6. The syringe is flushed several times by alternately filling the syringe with carrier gas and displacing the contents into the flow stream, without removing the syringe from the septum. Carrier flow is maintain through the cartridge for approximately 5 minutes after injection.

13.3 Preparation of Spiked Traps Using Permeation or Diffusion tubes

13.3.1 A flowing stream of inert gas containing known amounts of each compound of interest is generated according to ASTM Method D3609(6). Note that a method of accuracy maintaining temperature within ± 0.1°C is required and the system generally must be equilibrated for at least 48 hours before use.

13.3.2 An accurately known volume of the standard gas stream (usually 0.1-1 liter) is drawn through a clean Tenax cartridge using the sampling system described in Section 10.2.1, or a similar system. However, if mass flow controllers are employed they must be calibrated for the carrier gas used in Section 13.3.1 (usually nitrogen). Use of air as the carrier gas for permeation systems is not recommended, unless the compounds of interest are known to be highly stable in air.

13.3.3 The spiked cartridges are then stored or immediately analyzed as in Section 11.2.4.

14. Performance Criteria and Quality Assurance

This section summarizes quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory. In many cases the specific QA procedures have been described within the appropriate section describing the particular activity (e.g. parallel sampling).

14.1 Standard Opreating Procedures (SOPs)

14.1.1 Each user should generate SOPs describing the following activities as they are performed in their laboratory:

1) assembly, calibration, and operation of the sampling system,
2) preparation, handling and storage of Tenax cartridges,
3) assembly and operation of GC/MS system including the thermal desorption apparatus and data system, and
4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by the laboratory personnel conducting the work.

14.2 Tenax Cartridge Preparation

14.2.1 Each batch of Tenax cartridges prepared (as described in Section 9) should be checked for contamination by analyzing one cartridge immediately after preparation. While analysis can be accomplished by GC/MS, many laboratories may chose to use GC/FID due to logistical and cost considerations.

14.2.2 Analysis by GC/FID is accomplished as described for GC/MS (Section 11) except for use of FID detection.

14.2.3 While acceptance criteria can vary depending on the components of interest, at a minimum the clean cartridge should be demonstrated to contain less than one fourth of the minimum level of interest for each component. For most compounds the blank level should be less than 10 nanograms per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory. If a cartridge does not meet these acceptance criteria the entire lot should be rejected.

14.3 Sample Collection

14.3.1 During each sampling event at least one clean cartridge will accompany the samples to the field and back to the laboratory, without being used for sampling, to serve as a field blank. The average amount of material found on the field blank cartridge may be subtracted from the amount found on the actual samples. However, if the blank level is greater than 25% of the sample amount, data for that component must be identified as suspect.

14.3.2 During each sampling event at least one set of parallel samples (two or more samples collected simultaneously) will be collected, preferably at different flow rates as described in Section 10.1. If agreement between parallel samples is not generally within $\pm$ 25% the user should collect parallel samples on a much more frequent basis (perhaps for all sampling points). If a trend of lower apparent concentrations with increasing flow rate is observed for a set

of parallel samples one should consider using a reduced flow rate and longer sampling interval if possible. If this practice does not improve the reproducibility further evaluation of the method performance for the compound of interest may be required.

14.3.3 Backup cartridges (two cartridges in series) should be collected with each sampling event. Backup cartridges should contain less than 20% of the amount of components of interest found in the front cartridges, or be equivalent to the blank cartridge level, whichever is greater. The frequency of use of backup cartridges should be increased if increased flow rate is shown to yield reduced component levels for parallel sampling. This practice will help to identify problems arising from breakthrough of the component of interest during sampling.

14.4 GC/MS Analysis

14.4.1 Performance criteria for MS tuning and mass calibration have been discussed in Section 11.2 and Table 2. Additional criteria may be used by the laboratory if desired. The following sections provide performance guidance and suggested criteria for determining the acceptability of the GC/MS system.

14.4.2 Chromatographic efficiency should be evaluated using spiked Tenax cartridges since this practice tests the entire system. In general a reference compound such as perfluorotoluene should be spiked onto a cartridge at the 100 nanogram level as described in Section 13.2 or 13.3. The cartridge is then analyzed by GC/MS as

described in Section 11.4. The perfluorotoluene (or other reference compound) peak is then plotted on an expanded time scale so that its width at 10% of the peak can be calculated, as shown in Figure 6. The width of the peak at 10% height should not exceed 10 seconds. More stringent criteria may be required for certain applications. The assymmetry factor (See Figure 6) should be between 0.8 and 2.0. The assymmetry factor for any polar or reactive compounds should be determined using the process described above. If peaks are observed that exceed the peak width or assymmetry factor criteria above, one should inspect the entire system to determine if unswept zones or cold spots are present in any of the fittings and is necessary. Some laboratories may chose to evaluate column performance separately by direct injection of a test mixture onto the GC column. Suitable schemes for column evaluation have been reported in the literature (7). Such schemes cannot be conducted by placing the substances onto Tenax because many of the compounds (e.g. acids, bases, alcohols) contained in the test mix are not retained, or degrade, on Tenax.

14.4.3 The system detection limit for each component is calculated from the data obtained for calibration standards. The detection limit is defined as

$$DL = A + 3.3S$$

where

DL is the calculated detection limit in nanograms injected.

A is the intercept calculated in Section 12.1.1 or 12.1.3.

S is the standard deviation of replicate determinations of the lowest level standard (at least three such determinations are required.

In general the detection limit should be 20 nanograms or less and for many applications detection limits of 1-5 nanograms may be required. The lowest level standard should yield a signal to noise ratio, from the total ion current response, of approximately 5.

14.4.4 The relative standard deviation for replicate analyses of cartridges spiked at approximately 10 times the detection limit should be 20% or less. Day to day relative standard deviation should be 25% or less.

14.4.5 A useful performance evaluation step is the use of an internal standard to track system performance. This is accomplished by spiking each cartridge, including blank, sample, and calibration cartridges with approximately 100 nanograms of a compound not generally present in ambient air (e.g. perfluorotoluene). The integrated ion intensity for this compound helps to identify problems with a specific sample. In general the user should calculate the standard deviation of the internal standard response for a given set of samples analyzed under identical tuning and calibration conditions. Any sample giving a value greater than $\pm$ 2 standard deviations from the mean (calculated

excluding that particular sample) should be identified as suspect. Any marked change in internal standard response may indicate a need for instrument recalibration.

REFERENCES

1. Krost, K. J., Pellizzari, E. D., Walburn, S. G., and Hubbard, S. A., "Collection and Analysis of Hazardous Organic Emissions", Analytical Chemistry, 54, 810-817, 1982.

2. Pellizzari, E. O. and Bunch, J. E., "Ambient Air Carcinogenic Vapors-Improved Sampling and Analytical Techniques and Field Studies", EPA-600/2-79-081, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1979.

3. Kebbekus, B. B. and Bozzelli, J. W., "Collection and Analysis of Selected Volatile Organic Compounds in Ambient Air", Proc. Air Pollution Control Assoc., Paper No. 82-65.2. Air Poll. Control Assoc., Pittsburgh, Pennsylvania, 1982.

4. Riggin, R. M., "Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air", EPA-600/4-83-027, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1983.

5. Walling, J. F., Berkley, R. E., Swanson, D. H., and Toth, F. J. "Sampling Air for Gaseous Organic Chemical-Applications to Tenax", EPA-600/7-54-82-059, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1982.

6. Annual Book of ASTM Standards, Part 11.03, "Atmospheric Analysis", American Society for Testing and Material, Philadelphia, Pennsylvania.

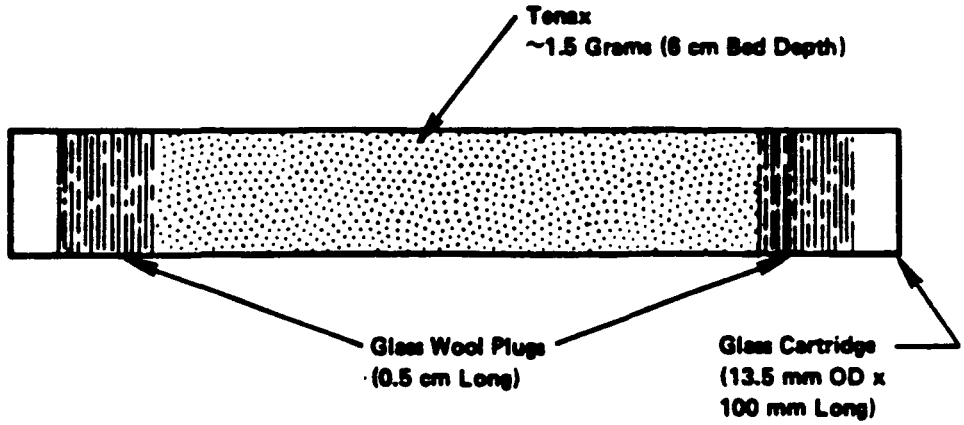
7. Grob, K., Jr., Grob, G., and Grob, K., "Comprehensive Standardized Quality Test for Glass Capillary Columns", J. Chromatog., 156, 1-20, 1978.

TABLE 1. RETENTION VOLUME ESTIMATES FOR COMPOUNDS ON TENAX

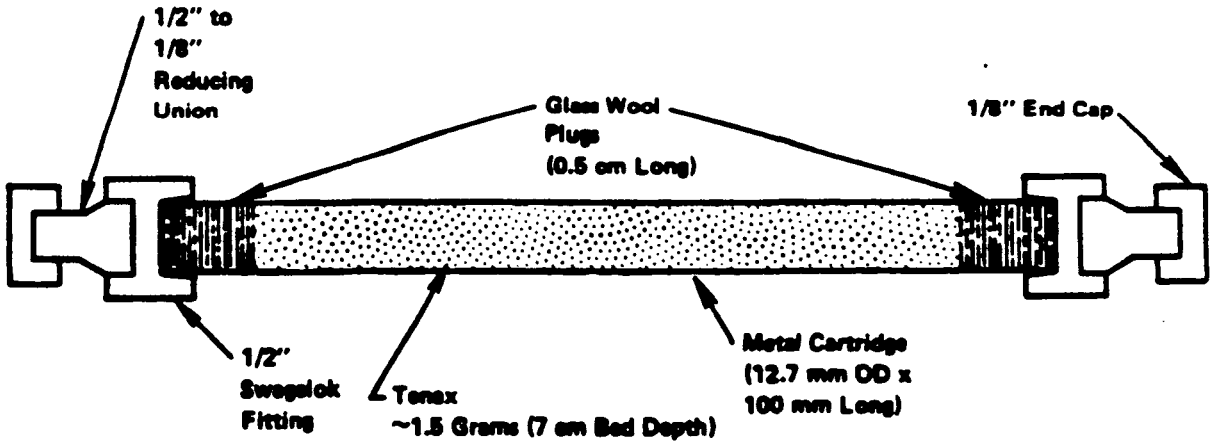
COMPOUND	ESTIMATED RETENTION VOLUME AT 100°F (38°C)-LITERS/GRAM
Benzene	19
Toluene	97
Ethyl Benzene	200
Xylene(s)	~ 200
Cumene	440
n-Heptane	20
1-Heptene	40
Chloroform	8
Carbon Tetrachloride	8
1,2-Dichloroethane	10
1,1,1-Trichloroethane	6
Tetrachloroethylene	80
Trichloroethylene	20
1,2-Dichloropropane	30
1,3-Dichloropropane	90
Chlorobenzene	150
Bromoform	100
Ethylene Dibromide	60
Bromobenzene	300

TABLE 2. SUGGESTED PERFORMANCE CRITERIA FOR RELATIVE ION ABUNDANCES FROM FC-43 MASS CALIBRATION

M/E	% RELATIVE ABUNDANCE
51	1.8 ± 0.5
69	100
100	12.0 ± 1.5
119	12.0 ± 1.5
131	35.0 ± 3.5
169	3.0 ± 0.4
219	24.0 ± 2.5
264	3.7 ± 0.4
314	0.25 ± 0.1

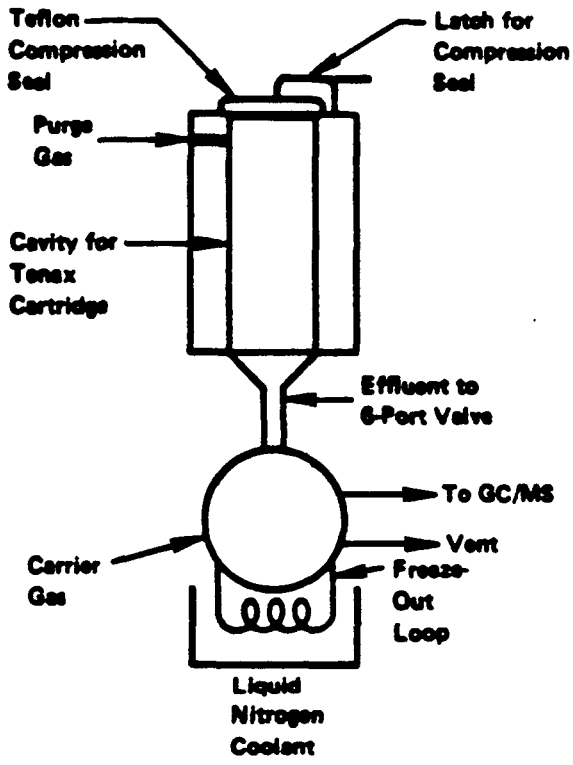


(a) Glass Cartridge

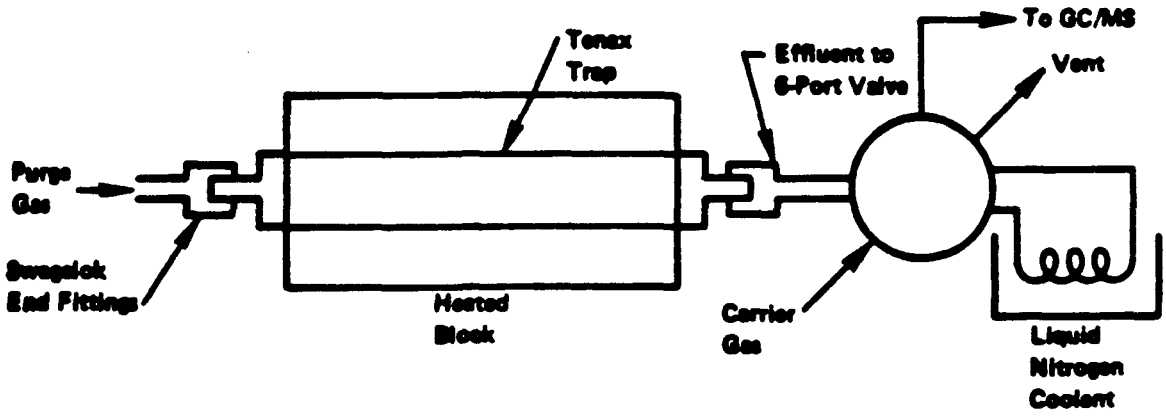


(b) Metal Cartridge

FIGURE 1. TENAX CARTRIDGE DESIGNS



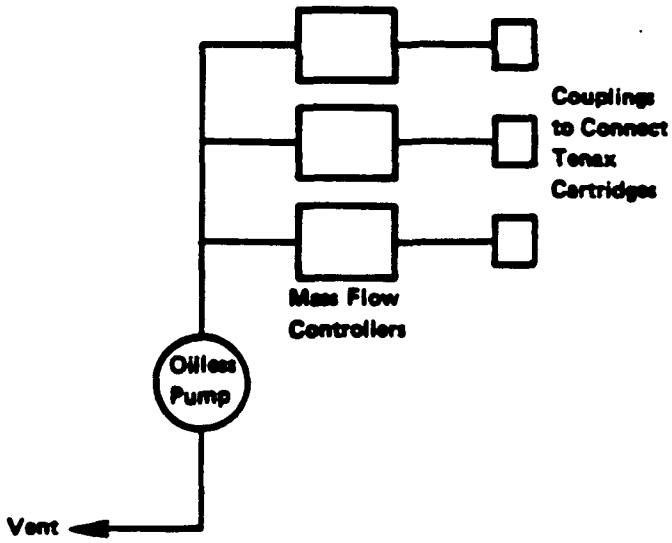
(a) Glass Cartridges (Compression Fit)



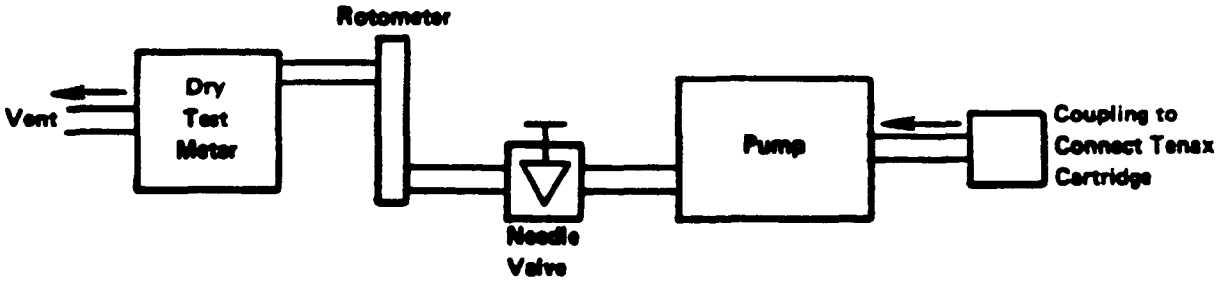
(b) Metal Cartridges (Swagelok Fittings)

FIGURE 2. TENAX CARTRIDGE DESORPTION MODULES

US4472



(a) Mass Flow Control



(b) Needle Valve Control

FIGURE 3. TYPICAL SAMPLING SYSTEM CONFIGURATIONS

SAMPLING DATA SHEET
(One Sample Per Data Sheet)

PROJECT:_____ DATE(S) SAMPLED:_____

SITE:_____ TIME PERIOD SAMPLED:_____

LOCATION:_____ OPERATOR:_____

INSTRUMENT MODEL NO:_____ CALIBRATED BY:_____

PUMP SERIAL NO:_____

SAMPLING DATA

Sample Number:_____

Start Time: _____ Stop Time: _____

Time	Dry Gas Meter Reading	Rotameter Reading	Flow Rate,*Q ml/Min	Ambient Temperature °C	Barometric Pressure, mmHg	Relative Humidity, %	Comments
1.							
2.							
3.							
4.							
N.							

Total Volume Data**

V_m = (Final - Initial) Dry Gas Meter Reading, or = _____ Liters

$$= \frac{Q_1 + Q_2 + Q_3 \ldots Q_N}{N} \times \frac{1}{1000 \times (\text{Sampling Time in Minutes})} = \_\_\_\_\_ \text{ Liters}$$

* Flowrate from rotameter or soap bubble calibrator (specify which).
** Use data from dry gas meter if available.

FIGURE 4. EXAMPLE SAMPLING DATA SHEET

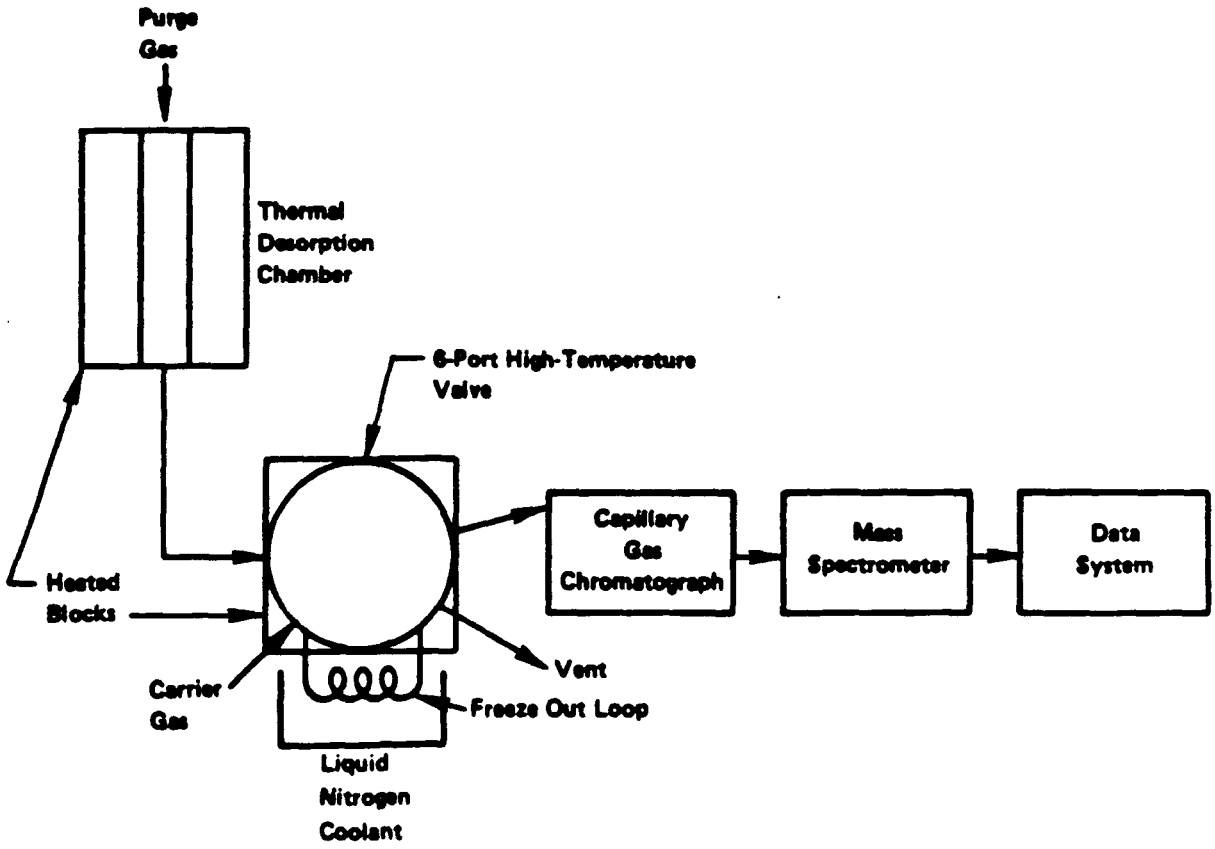
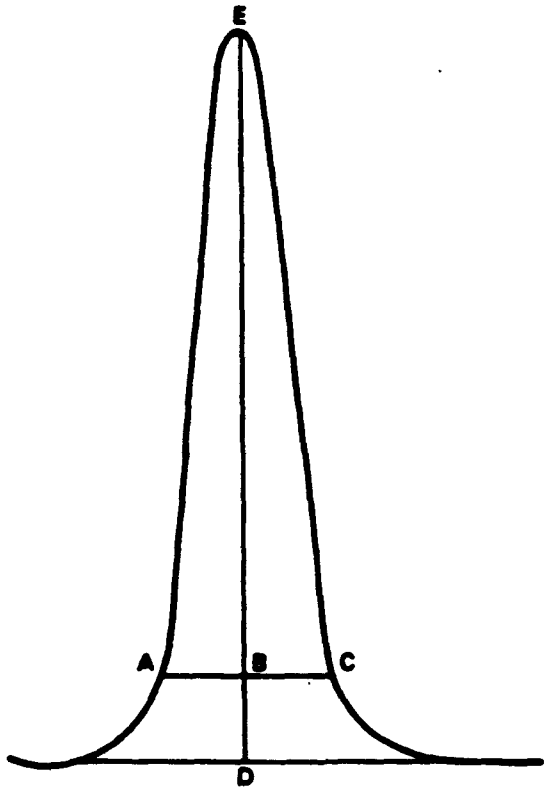


FIGURE 5. BLOCK DIAGRAM OF ANALYTICAL SYSTEM



$$\text{Asymmetry Factor} = \frac{BC}{AB}$$

Example Calculation:

Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Asymmetry Factor = $\frac{12}{11} = 1.1$

FIGURE 6. PEAK ASYMMETRY CALCULATION

ATTACHMENT 2

PROTOCOL FOR PERFORMANCE EVALUATION

16.4.5 A useful performance evaluation step is the use of an internal standard to track system performance. This is accomplished by spiking each cartridge, including blank, sample, and calibration cartridges with approximately 100 nanograms of a compound not generally present in ambient air (e.g. perfluorotoluene). The integrated ion intensity for this compound helps to identify problems with a specific sample. In general the user should calculate the standard deviation of the internal standard response for a given set of samples analyzed under identical tuning and calibration conditions. Any sample giving a value greater than ± 2 standard deviations from the mean (calculated

excluding that particular sample) should be identified as suspect. Any marked change in internal standard response may indicate a need for instrument recalibration.

ATTACHMENT 3

PROTOCOL FOR INSTRUMENT CALIBRATION

11.2 Instrument Calibration

11.2.1 Tuning and mass standarization of the MS system is performed according to manufacturer's instructions and relevent information from the user prepared SOP. Perfluorotributylamine should generally be employed for this purpose. The material is introduced directly into the ion source through a molecular leak. The instrumental parameters (e.g. lens voltages, resolution, etc.) should be adjusted to give the relative ion abundances shown in Table 2 as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source may require cleaning according to manufacturer's instructions. In the event that the user's instrument cannot achieve these relative ion abundances, but is otherwise operating properly, the user may adopt another set of relative abundances as performance criteria.

However, these alternate values must be repeatable on a day-to-day basis.

11.2.2 After the mass standarization and tuning process has been completed and the appropriate values entered into the data system the user should then calibrate the entire system by introducing known quantities of the standard components of interest into the system. Three alternate procedures may be employed for the calibration process including 1) direct syringe injection of dilute vapor phase standards, prepared in a dilution bottle, onto the GC column, 2) injection of dilute vapor phase standards into a carrier gas stream directed through the Tenax cartridge, and 3) introduction of permeation or diffusion tube standards onto a Tenax cartridge. The standards preparation procedures for each of these approaches are described in Section 13. The following paragraphs describe the instrument calibration process for each of these approaches.

11.2.3 If the instrument is to be calibrated by direct injection of a gaseous standard, a standard is prepared in a dilution bottle as described in Section 13.1. The GC column is cooled to -70°C (or, alternately, a portion of the column inlet is manually cooled with liquid nitrogen). The MS and data system is set up for acquisition as described in the relevant user SOP. The ionization filament should be turned off during the initial 2-3 minutes of the run to allow oxygen and other highly volatile components to elute. An appropriate volume (less than 1 ml) of the gaseous standard is injected onto the GC system using an accurately calibrated gas tight syringe.

The system clock is started and the column is maintained at -70°C (or liquid nitrogen inlet cooling) for 2 minutes. The column temperature is rapidly increased to the desired initial temperature (e.g. 30°C The temperature program is started at a consistent time (e.g. four minutes) after injection. Simultaneous the ionization filament is turned on and data acquisit is initiated. After the last component of interest has eluted acquisition is terminated and the data is proces as described in Section 11.2.5. The standard injection process is repeated using different standard volumes as desired.

11.2.4 If the system is to be calibrated by analysis of spiked Tenax cartridges a set of cartridges is prepared as described in Sections 13.2 or 13.3. Prior to analysis the cartridges are stored as described in Section 9.3. If glass cartridges (Figure are employed care must be taken to avoid direct contact, as described earlier. The GC column is cooled to -70°C, the collection loop is immersed in liquid nitrogen and the desorption module is maintained at 250°C. The inlet valve is placed in t desorb mode and the standard cartridge is placed in the desorption module, making certain that no leaks of purge gas occurs. The cartridge is purged for 10 minutes and then the inlet valve is placed in the inject mode and the liquid nitrogen source remo from the collection trap. The GC column is maintai at -70°C for two minutes and subsequent steps are a described in 11.2.3. After the process is complete cartridge is removed from the desorption module and stored for subsequent use as described in Section 1

11.2.8 Data processing for instrument calibration involves determining retention times, and integrated characteristic ion intensities for each of the compounds of interest. In addition, for at least one chromatographic run, the individual mass spectra should be inspected and compared to reference spectra to ensure proper instrumental performance. Since the steps involved in data processing are highly instrument specific, the user should prepare a SOP describing the process for individual use. Overall performance criteria for instrument calibration are provided in Section 14. If these criteria are not achieved the user should refine the instrumental parameters and/or operating procedures to meet these criteria.

ATTACHMENT 4

CARTRIDGE CONSTRUCTION AND PREPARATION

9. Cartridge Construction and Preparation

9.1 Cartridge Design

9.1.1 Several cartridge designs have been reported in the literature (1-3). The most common (1) is shown in Figure 1a. This design minimizes contact of the sample with metal surfaces, which can lead to decomposition in certain cases. However, a disadvantage of this design is the need to rigorously avoid contamination of the outside portion of the cartridge since the entire surface is subjected to the purge gas stream during the desorption process.

Clean polyester gloves must be worn at all times when handling such cartridges and exposure of the open cartridge to ambient air must be minimized.

9.1.2 A second common type of design (3) is shown in Figure 1b. While this design uses a metal (stainless steel) construction, it eliminates the need to avoid direct contact with the exterior surface since only the interior of the cartridge is purged.

9.1.3 The thermal desorption module and sampling system must be selected to be compatible with the particula cartridge design chosen. Typical module designs are shown in Figures 2a and b. These designs are suitable for the cartridge designs shown in Figures 1a and 1b, respectively.

9.2 Tenax Purification

9.2.1 Prior to use the Tenax resin is subjected to a series of solvent extraction and thermal treatment steps. The operation should be conducted in an area where levels of volatile organic compounds (other than the extraction solvents used) are minimized.

9.2.2 All glassware used in Tenax purification as well as cartridge materials should be thoroughly cleaned by water rinsing followed by an acetone rinse and dried in an oven at 250°C.

9.2.3 Bulk Tenax is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in the soxhlet extraction apparatus and extracted sequentially with methanol and then pentane for 16-24 hours (each solvent) at approximately 6 cycles/hour. Glasswool for cartidge preparation should be cleaned in the same manner as Tenax.

9.2.4 The extracted Tenax is immediately placed in an open glass dish and heated under an infrared lamp for two

hours in a hood. Care must be exercised to avoid over heating of the Tenax by the infrared lamp. The Tenax is then placed in a vacuum oven (evacuated using a water aspirator) without heating for one hour. An inert gas (helium or nitrogen) purge of 2-3 ml/minute is used to aid in the removal of solvent vapors. The oven temperature is then increased to 110°C, maintaining inert gas flow and held for one hour. The oven temperature control is then shut off and the oven is allowed to cool to room temperature Prior to opening the oven, the oven is slightly pressurized with nitrogen to prevent contamination with ambient air. The Tenax is removed from the oven and sieved through a 60/80 mesh sieve (acetone rinsed and oven dried) into a clean glass vessel. If the Ten is not to be used immediately for cartridge preparatio it should be stored in a clean glass jar having a Teflon-lined screw cap and placed in a desiccator.

9.3 Cartridge Preparation and Pretreatment

9.3.1 All cartridge materials are pre-cleaned as described in Section 9.2.2. If the glass cartridge design show in Figure 1a is employed all handling should be conducted wearing polyester gloves.

9.3.2 The cartridge is packed by placing a 0.5-1cm glass-wool plug in the base of the cartridge and then filling the cartridge to within approximately 1 cm of the top. A 0.5-1cm glasswool plug is placed in the top of the cartridge.

9.3.3 The cartridges are then thermally conditioned by heating for four hours at 270°C under an inert gas (helium) purge (100 - 200 ml/min).

9.3.4 After the four hour heating period the cartridges are allowed to cool. Cartridges of the type shown in Figure 1a are immediately placed (without cooling) in clean culture tubes having Teflon-lined screw caps with a glasswool cushion at both the top and the bottom. Each tube should be shaken to ensure that the cartridge is held firmly in place. Cartridges of the type shown in Figure 1b are allowed to cool to room temperature under inert gas purge and are then closed with stainless steel plugs.

9.3.5 The cartridges are labeled and placed in a tightly sealed metal can (e.g. paint can or similar friction top container). For cartridges of the type shown in Figure 1a the culture tube, not the cartridge, is labeled.

9.3.6 Cartridges should be used for sampling within 2 weeks after preparation and analyzed within two weeks after sampling. If possible the cartridges should be stored at -20°C in a clean freezer (i.e. no solvent extracts or other sources of volatile organics contained in the freezer).

ATTACHMENT 5

CARTRIDGE CONTAMINATION CHECK

14.2 Tenax Cartridge Preparation

14.2.1 Each batch of Tenax cartridges prepared (as described in Section 9) should be checked for contamination by analyzing one cartridge immediately after preparation. While analysis can be accomplished by GC/MS, many laboratories may chose to use GC/FID due to logistical and cost considerations.

14.2.2 Analysis by GC/FID is accomplished as described for GC/MS (Section 11) except for use of FID detection.

14.2.3 While acceptance criteria can vary depending on the components of interest, at a minimum the clean cartridge should be demonstrated to contain less than one fourth of the minimum level of interest for each component. For most compounds the blank level should be less than 10 nanograms per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory. If a cartridge does not meet these acceptance criteria the entire lot should be rejected.

APPENDIX C

CALIBRATION AND MAINTENANCE PROCEDURE FOR ANALYTICAL FIELD EQUIPMENT

EQUIPMENT AND INSTRUMENT CALIBRATION AND MAINTENANCE, GENERAL REQUIREMENTS

1.0 INTRODUCTION

The general guidelines for calibrating and maintaining instruments and monitoring equipment are presented in this document.

2.0 CALIBRATION AND MAINTENANCE PROCEDURES

Calibration and maintenance procedures are documented for each piece of equipment affecting quality. Calibration and maintenance procedures are developed based on manufacturer's specifications and are retained in the Site Investigation Procedures Manual. These procedures include, but are not limited to:

1. Equipment identification (name) and description.
2. Equipment specifications.
3. Calibration and/or maintenance schedule.
4. Equipment necessary to accomplish calibration (where applicable).
5. Procedure for calibration and/or maintenance.

3.0 CALIBRATION LABEL

Instruments requiring calibration and/or maintenance have a prominently displayed sticker containing the following information:

1. Date of calibration and/or maintenance.
2. Next due date for calibration and/or maintenance.
3. Initials of person performing calibration and/or maintenance.
4. Span gas and concentration(s) (if applicable).
5. Span or sensitivity setting (if applicable).

4.0 EQUIPMENT LOG BOOK

An equipment log book is issued to record the life history of each measuring and testing device used in activities affecting quality. This book is a three ring binder in which individual records for each piece of equipment are maintained. A form such as F6101 or a reasonable facsimile should be used to maintain the calibration and maintenance record. The record should include:

1. Equipment identification (name) and control number.
2. Date of calibration and/or maintenance.
3. Condition of equipment.
4. Activity performed on instrument (calibration and/or maintenance).
5. Adjustments made and accuracy of equipment prior to and following calibration (where applicable).
6. Record of equipment failure or inability to meet specifications (where applicable).
7. Initials of person performing calibration/maintenance.
8. Next due date for calibration and/or maintenance.

5.0 CALIBRATION/MAINTENANCE FORM

An instrument specific calibration/maintenance form will be developed to record data relating to each individual calibration/maintenance event. A single form will be used for each calibration/maintenance event. In addition to the data recorded in the calibration/maintenance log, the following items should also be included in the instrument specific form (where applicable).

1. Calibration calculations and curves.
2. Span gas type and concentrations.
3. Span or sensitivity range settings.
4. Specifics on repairs and parts replaced, added, or removed.

5. Instrument's overall condition.

6.0 FIELD CALIBRATION

As part of normal field operations, some instruments require calibration prior to, during, and/or after field use. This field operation calibration should remain separate from pre-field calibrations and should not be used as a substitute for standard calibration activities. Field calibration should be recorded in field log books or on field forms as part of the normal field data collection process. Field calibration records should not be included in the history log.

7.0 INSTRUMENTS NOT IN COMPLIANCE

If the calibration schedule is not adequately maintained, or if accuracy as reported in specifications cannot be attained for a specific instrument, that instrument is labelled "HOLD" and is unavailable for use until it is repaired and specifications are attained.

CALIBRATION AND MAINTENANCE PROCEDURE
YSI MODEL 33 S-C-T METER

1.0 INTRODUCTION

This procedure presents steps to calibrate and maintain the YSI Model 33 S-C-T meter. Operation principles, procedures, and equipment specifications are presented in Procedure 5617002 and are not repeated here.

2.0 CALIBRATION

2.1 Temperature

2.1.1 Temperature Knob Setting

It is possible for the temperature knob to become loose or slip from its normal position. In an emergency, the dial can be repositioned. It must be emphasized that this is an emergency procedure only and that the instrument should be returned to the factory for proper recalibration - at the earliest opportunity.

To recalibrate the temperature setting:

1. Red line instrument and then place probe in sample of known conductivity.
2. Read and record the temperature and conductivity of the solution using appropriate settings. Leave probe in solution.
3. Determine the salinity of the solution by running a line vertically on Figure 1 until it intersects the appropriate 'C line. From this intersection, extend a line horizontally to the left edge of the graph (Figure 1). This determines the salinity of the sample.

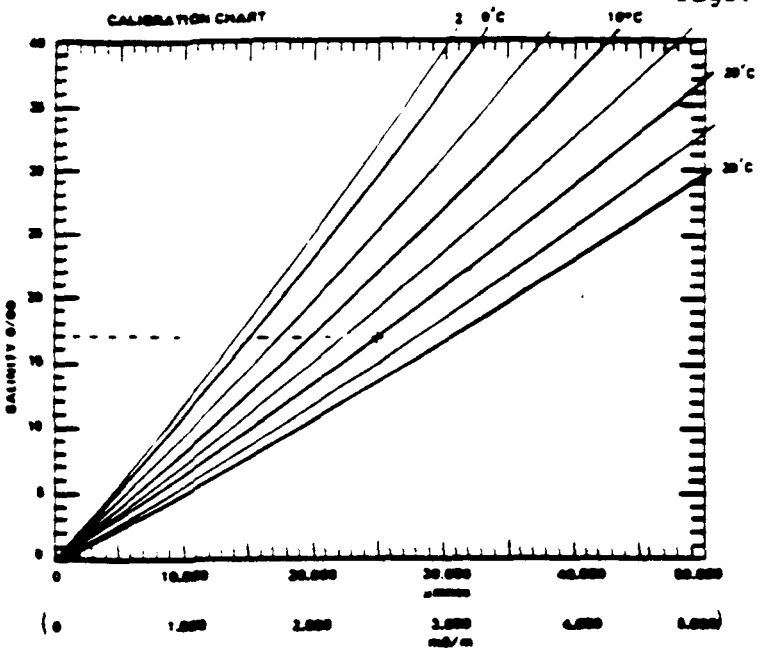


Figure 1. Calibration Chart for Resetting Temperature Knob

4. Remove the °C knob switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in step 3.

5. Switch to TEMPERATURE. If this temperature is the same as step 2, continue. If not, repeat steps 1 through 5.

6. Place the knob on the control shaft - without turning the control shaft - with the pointer at the same temperature as the meter reading. Tighten both sets of screws securely. Care must be taken at this step so that the shaft setting is not moved.

7. Return the instrument to the factory at the earliest opportunity.

2.1.2 Tempertaure Probe/Instrument

To check the accuracy of the Probe/Instrument temperature readings:

1. Place NBS traceable thermometer in solution with thermometer and probe.

2. Place instrument in temperature mode after zeroing and red lining the instrument.

3. After five minutes, compare temperature of thermometer and instrument. If the instrument varies by ±1C, the instrument should be returned to the factory for calibration and maintenance.

2.2 Probe Cell Calibration

The YSI #3300 Series Cells are calibrated to absolute accuracy of ± 1.5 percent based on a standard solution of 0.01 demol KCl. To prepare this solution:

1. In a one liter flask, dissolve 0.745 grams of pure dry KCl until the solution is one kilogram in weight.

2. Use Table 1 and the temperature of the water to determine the conductivity of the solution just prepared. Note: Table 1 shows conductivity as if the distilled water was nonconductive. Since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

3. Place probe in solution and measure conductivity. The conductivity of the solution plus the conductivity of the distilled water should not vary from the meter reading by ± 1.5%. If the reading is greater than 1.5%, clean the probe and then recheck the conductivity. If after cleaning it is not possible to measure the conductivity of the calibration solution within ± 1.5%, the probe and instrument should be returned to the manufacturer for calibration and maintenance.

3.0 MAINTENANCE

3.1 Batteries

The batteries should be replaced either (1) when it is not possible to red line the instrument, (2) after 200 hours of operation, or (3) every 6 months to reduce the danger of corrosion due to leaky batteries.

To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The positive (+ button) end must go on red.

Use two "D" size alkaline flashlight cells (Eveready E95 or equivalent).

3.2 Probe

3.2.1 Cleaning

When the cell test indicates low readings, the probable cause is dirty electrodes. Hard water deposits, oils, and organic matter are the most likely contaminants.

TABLE 1 - CELL CALIBRATION DATA

Temperature (°C)	Conductivity (umhos/cm)
15	1141.5
16	1167.5
17	1193.6
18	1219.9
19	1246.4
20	1273.0
21	1299.7
22	1326.6
23	1353.6
24	1380.8
25	1408.1
26	1436.5
27	1463.2
28	1490.9
29	1518.7
30	1546.7

For convenient normal cleaning, soak the electrodes for 5 minutes with a locally available bathroom tile cleaner such as: "Rally, Tile, Porcelain, and Chrome Cleaner"; Johnson Wax "Envy, Instant Cleaner"; or Lysol Brand "Basin, Tub, Tile Cleaner".

For storage cleaning, a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol, and 1 part HCl can be used.

Always rinse the probe in distilled water after cleaning and before storage.

CAUTION: Do not touch the electrodes inside the probe. Platinum black is very soft and can be scraped off.

If cleaning does not restore the probe performance, re-platinizing is required.

3.2.2 Probe Replatinizing

1. Equipment required:
   a. YSI #3140 Platinizing Solution, 2 fluid ounce (3% platinum chloride dissolved in 0.025% lead acetate solution)
   b. YSI Model 33 meter
   c. 50 ml glass beaker or equivalent
   d. Distilled water
2. Procedure
   a. Clean probe as in section 3.2.1 - either method

b. Place the cell in the beaker and add sufficient YSI #3140 solution to cover the electrodes. Do not cover the top of the probe

c. Plug the probe into the Model 33 and switch to the X100 scale to platinize the electrode

d. Move the probe slightly to obtain the highest meter reading and continue platinizing for the appropriate time shown below:

Meter Reading (umhos/cm)	Time (minutes)
30,000	5
25,000	6
20,000	8
15,000	11
10,000	16

e. After the elapsed time, remove the probe and rinse in distilled water.

f. Return the solution to its container. Two ounces of solution should be sufficient for 50 treatments.

3.2.3 Storage

It is best to store conductivity cells in deionized water. Cells stored in water require less frequent platinization. Any cell that has been stored dry should be soaked in deionized water for 24 hours before use.

CALIBRATION AND MAINTENANCE PROCEDURES HAAKEBUCHLER pH STICK

1.0 INTRODUCTION

This procedure presents the steps for calibrating and maintaining the HaakeBuchler pH Stick. Instrument operation principles and procedures and specifications are presented in Procedure 5617003.

2.0 CALIBRATION

2.1 Calibration Solutions

The instrument requires distilled water, a pH 7 buffer solution, and a pH 4 buffer solution for calibration. To prepare the buffer solutions, dissolve the buffer powders provided with the instrument into the volume of distilled water specified on the buffer powder packets. (Note: the manufacturer does not specify whether buffer and pH 4 solutions, other than that provided, may be used as substitute solutions).

The pH of the buffer and pH 4 solutions will vary with the temperature of the solution. Use the table below to determine solution pH based on temperature.

Temp	0°C	10°C	20°C	25°C	30°C	40°C	50°C
pH 4	4.00	4.00	4.00	4.01	4.02	4.04	4.06
pH 7	7.11	7.06	7.01	7.00	6.98	6.97	6.97

2.2 Calibration Procedure

The instrument requires calibration in the field prior to each use. However, as a check of proper instrument function, the instrument should be periodically calibrated in the laboratory,

particularly if the instrument has been stored for an extended period without use.

To calibrate the instrument:

1. Remove the protective sheath and rinse the electrode in distilled water.
2. Place the electrode in the pH 7 buffer solution, depress the white operation button below the LCD display and allow the reading to stabilize.
3. Adjust pH 7 control using the tool on the end of the protective sheath. The pH 7 control is the upper most white control on the right side of the instrument. Adjust the pH control until the meter reads pH 7.
4. Rinse the electrode in distilled water.
5. Place the electrode in pH 4 solution, depress the white operation button, and allow the reading to stabilize.
6. Adjust the slope control (white control below pH 7 control on the right side of the instrument) until the meter reads the correct value of the pH 4 solution.
7. Rinse the probe in distilled water.
8. Repeat steps 2 through 7.
9. Record calibration on the instrument log form.
10. Store instrument properly.

3.0 MAINTENANCE

3.1 Storage

To maintain high accuracy and to obtain a long electrode life, the pH stick must be stored correctly when not in use. Always rinse the electrode in distilled water before replacing it in its protective sheath. The electrode must not be let to dry out.

The absorbent pad at the bottom of the sheath must be kept saturated with a pH 7 buffer solution. If this is not available, distilled water can be used as a temporary measure. Replace distilled water with buffer solution at the earliest possible opportunity. Always place buffer (or distilled water) into sheath following each use.

To retain accuracy and speed of response, the insulation of the connectors on the electrode and the body must be kept clean and dry. This is best assured by not unnecessarily removing the electrode from the body.

When not in use, place the pH stick in the wallet provided and store in a dry place.

3.2 Electrode Cleaning

If rinsing the electrode in distilled water is not deemed sufficient to clean the electrode, it can be cleaned in a N/10 HCl acid solution. Following cleaning in the acid, the electrode should be soaked in a pH 7 buffer solution for 24 hours before rinsing. Record cleaning on instrument's log form.

3.3 Battery

Normal battery life is in excess of 200 hours of continuous use. Cells should be replaced at 2 year intervals or earlier if exhausted (voltage per cell of less than 1.35V). Replacement cells must be mercury type V312H or direct equivalent. When refitting cells, make sure they are refitted in the manner illustrated on the battery housing.

CALIBRATION PROCEDURE FOR THE HNu PI 101

1.0 INTRODUCTION

1.1 Content

This procedure presents the steps required to calibrate the HNu Model PI 101 photoionization analyzer. This instrument should be calibrated after each field use or prior to each field use if the instrument has not been calibrated during the previous 14 calendar days. The principle of detection and operating procedures are described in Procedure 5607001. This procedure presents calibration steps only.

1.2 Equipment

- Calibration Gas (2 ranges)

  Low range 0-20 ppm and mid range 20-200 ppm Isobutylene gas for standard field operation when contaminants are unknown or a mixture of gases is present. Isobutylene is the gas used for general calibration because of the instrument's relatively high sensitivity to it and the non-toxic nature of the gas.

  Note: A specialty gas may be required if a single atmospheric contaminant is present and the contaminant has a sensitivity different from that of the calibration gas. See procedure for 5607001 for a discussion on specialty calibration.

- Tubing and fittings (See Figure 1).
- Rotometer or bubble flow meter.
- Calibration Form F6264.
- Table 1 for ionization potentials for compounds of interest.

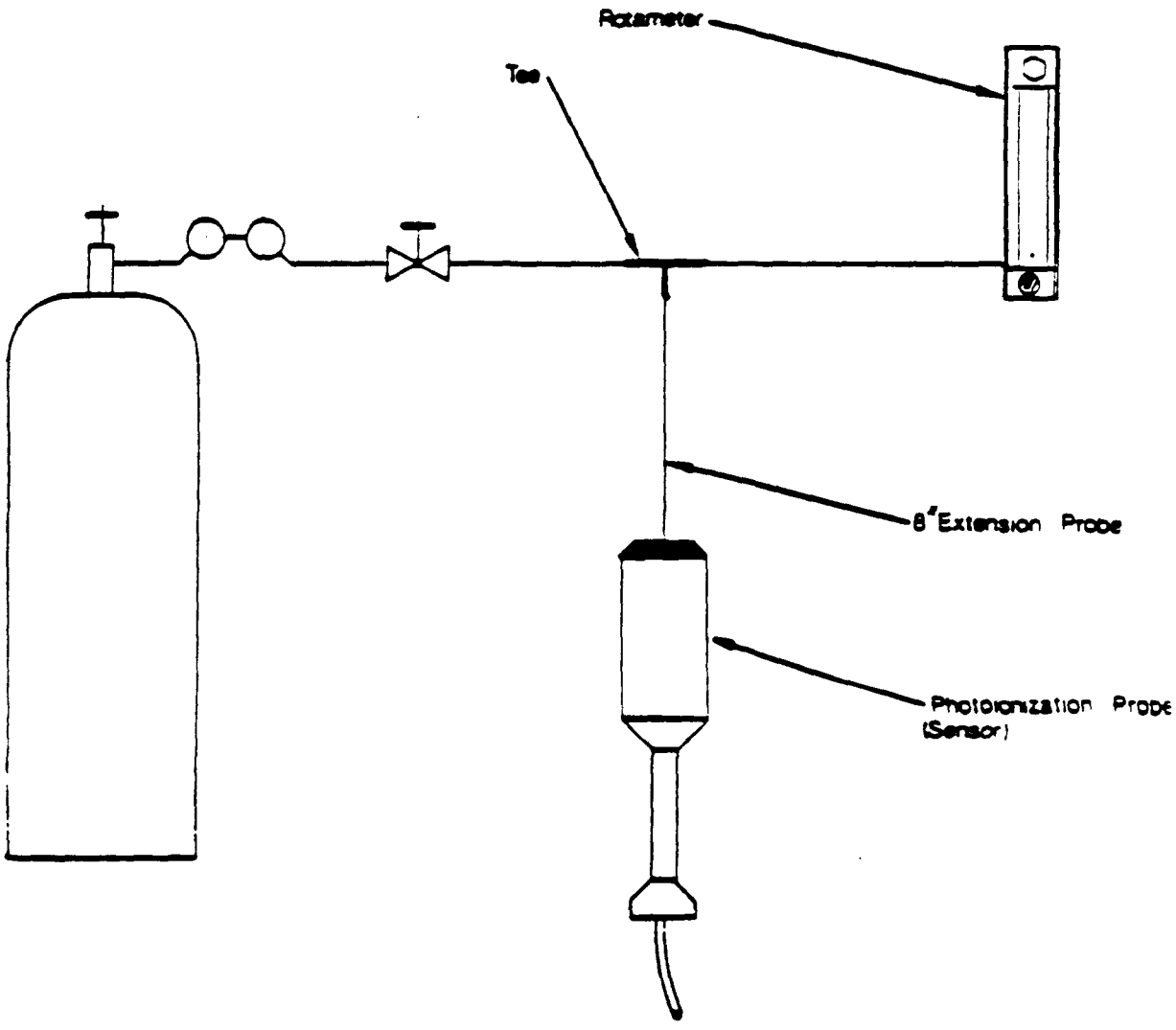


FIGURE 1 RECOMMENDED CALIBRATION PROCEDURE FOR PHOTOIONIZATION ANALYZER

TABLE 1 RELATIVE SENSITIVITIES FOR VARIOUS GASES
(10.2 eV Lamp)

Species	Photoionization Sensitivity*
p-xylene	11.4
m-xylene	11.2
benzene	10.0 (reference standard)
toluene	10.0
diethyl sulfide	10.0
diethyl amine	9.9
styrene	9.7
trichloroethylene	8.9
carbon disulfide	7.1
isobutylene	7.0
acetone	6.3
tetrahydrofuran	6.0
methyl ethyl ketone	5.7
methyl isobutyl ketone	5.7
cyclohexanone	5.1
naptha (85% aromatics)	5.0
vinyl chloride	5.0
methyl isocyanate	4.5
iodine	4.5
methyl mercaptan	4.3
dimethyl sulfide	4.3
allyl alcohol	4.2
propylene	4.0
mineral spirits	4.0
2,3-dichloropropene	4.0
cyclohexene	3.4
crotonaldehyde	3.1
acrolein	3.1
pyridine	3.0
hydrogen sulfide	2.8
ethylene dibromide	2.7
n-octane	2.5
acetaldehyde oxime	2.3
hexane	2.2
phosphine	2.0
heptane	1.7
allyl chloride (3-chloropropene)	1.5
ethylene	1.0
ethylene oxide	1.0
acetic anhydride	1.0
a-pinene	0.7
dibromochloropropane	0.7
epichlorohydrin	0.7
nitric oxide	0.6

TABLE 1 RELATIVE SENSITIVITIES FOR VARIOUS GASES
(10.2 eV Lamp) (Continued)

Species	Photoionization Sensitivity*
b-pinene	0.5
citral	0.5
ammonia	0.3
acetic acid	0.1
nitrogen dioxide	0.02
methane	0.0
acetylene	0.0

*Expressed in ppm (v/v).

2.0 CALIBRATION PROCEDURE

2.1 CDM employs a two-point standardization procedure to facilitate proper instrument calibration over the 0-20 ppm and 20-200 ppm operating ranges. Two distinct mixtures of the calibration gas (isobutylene) in air are used. Each mixture should give a 3/4 scale deflection in its respective operating range.

2.2 Instrument Setup.

2.2.1 Remove Instrument cover by pulling up on the side straps.

2.2.2 Prior to calibration, check the function switch (Figure 2) on the control panel to make sure it is in the OFF-positon. The probe nozzle, is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.

2.2.3 Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.

2.2.4 Attach probe cable to instrument box by inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.

2.2.5 Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light

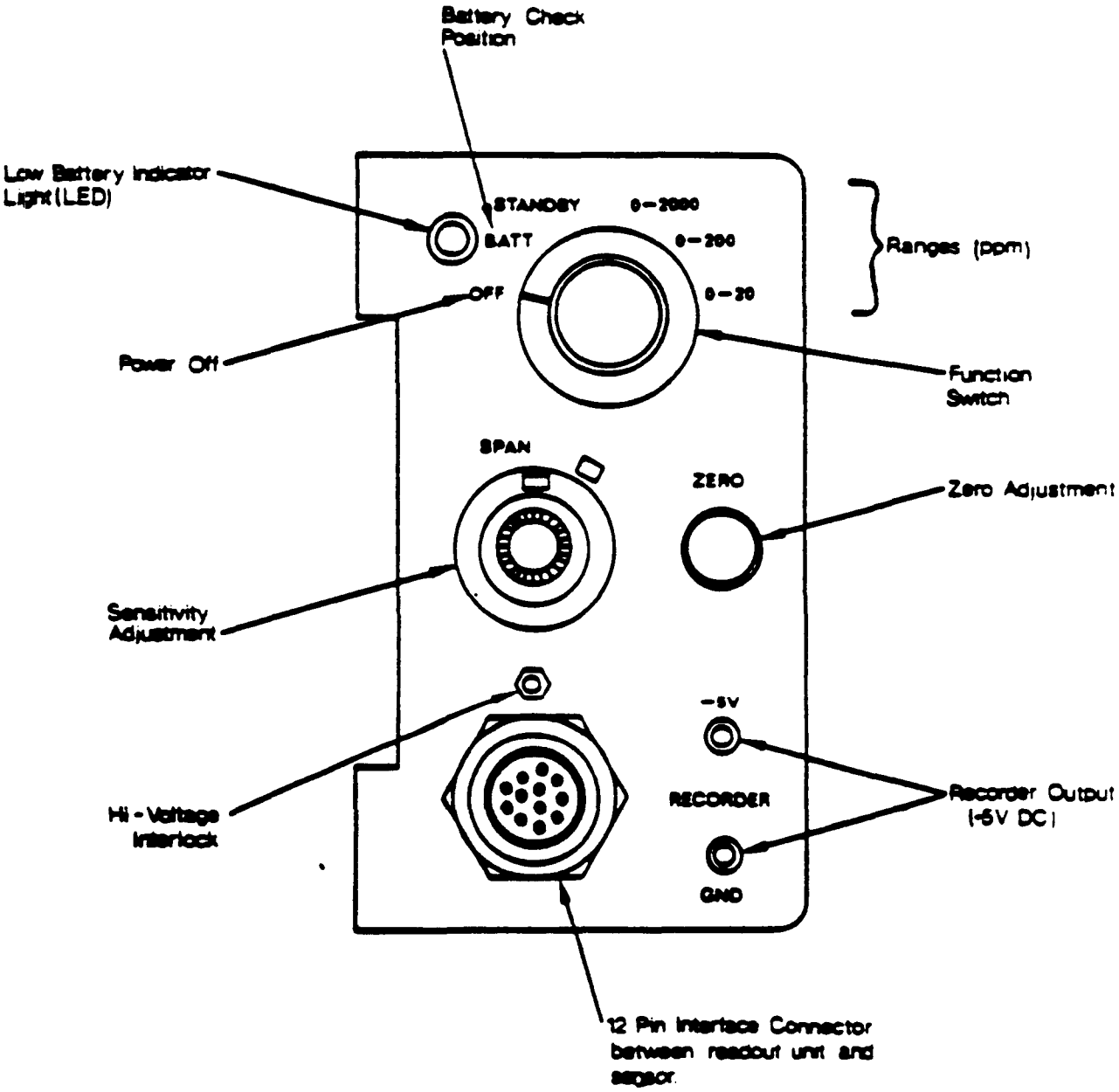


FIGURE 2 CONTROL PANEL FEATURES

US4509

comes on, the instrument should be recharged prior to making any measurements. Implement step 3.0 to recharge battery.

2.2.6 Turn the function switch to the ON position. In this position, the UV light source should be on. To verify, gaze at the end of the probe for a purple glow. Do not look directly at the lamp itself. If the lamp does not come on refer to maintenance step 4.1.2.

2.2.7 To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: no zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

2.3 Calibration Steps

2.3.1 Insert one end of T tube (Figure 1) into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubblemeter) attached.

2.3.2 Set the function switch in the 0-200 ppm range.

2.3.3 Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotometer. The instrument will draw in the volume required for detection with the rotometer indicating excess flow.

2.3.4 Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder).

2.3.5 Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following step 2.3.7.

2.3.6 Record on form F6264 all original and readjusted settings as specified in the form.

2.3.7 Next, set the function switch to the 0-20 ppm. Remove the mid range (20-200 ppm) calibration gas cylinder and attach the low range (0-20 ppm) calibration gas cylinder as described above.

2.3.8 Do not adjust the span potentiometer. The observed reading should be $\pm$ 3ppm of the concentration specified for the low range calibration gas. If this is not the case, recalibrate the mid range scale repeating procedures 3.3.1 to 3.2.7 above. If the low range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window following step 4.1.3. When the observed reading is within the required tolerances, the instrument is fully calibrated.

2.3.9 Complete forms F6264 and F6265 for the respective instrument being calibrated.

3.0 BATTERY RECHARGING

3.1 Place plug on end of charger cable into jack on left side of instrument case

3.2 Plug charger into 120V AC supply.

3.3 To ensure that charger is functioning, turn the function switch to the battery check position. The meter should go upscale if the charger is working correctly and correctly inserted.

3.4 The battery is completely charged overnight (ca, 14 hours).

3.5 When disconnecting charger, remove from 120 V AC before removing mini phone plug.

4.0 TROUBLE SHOOTING AND MAINTENANCE

4.1 General Fault Determination and Correction

4.1.1 Battery level is low. Recharge if necessary implementing step 3.0. If the battery will not recharge it will have to be replaced.

4.1.2 UV lamp function. Gaze at sample inlet when mode switch is on an instrument function position and observe for purple glow of lamp. If the lamp does not glow in any of the three instrument function positions, it may be burned out and will have to be replaced. To replace the lamp:

US4512

1. Turn the function switch to the off position and disconnect the probe connector from the readout unit.

2. Remove the exhaust screw found near the base of the probe (Figure 3).

3. Grasp the end cap in one hand and the probe shell in the other and gently pull to separate the end cap and lamp housing from the shell.

4. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing. Care must be taken so that the ion chamber does not fall out of the end cap and the lamp does not slide out of the lamp housing.

5. Turn the end cap over in your hand and tap on the top of it; the ion chamber should fall out of it.

6. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.

7. Replace lamp with one of same energy source as the one removed by sliding it into the housing. Note: the amplifier board and instrument circuitry are calibrated for one light energy source. Insertion of a lamp of a different energy level will produce false instrument readings.

8. Place the ion chamber on top of the lamp housing, checking to ensure that the contacts are aligned.

9. Place the end cap on top of the ion chamber and replace the two screws. The screws should be tightened only enough to seal the "O" ring. Do not overtighten.

10. Line up the pins on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. Do not force the assembly as it only fits one way.

11. Replace and tighten the exhaust screw.

12. Reconnect the 12 pin connector and turn instrument mode switch to a function position. Check for glow of lamp. If lamp still does not function the instrument has an electrical short or other problem that will have to be corrected at the factory.

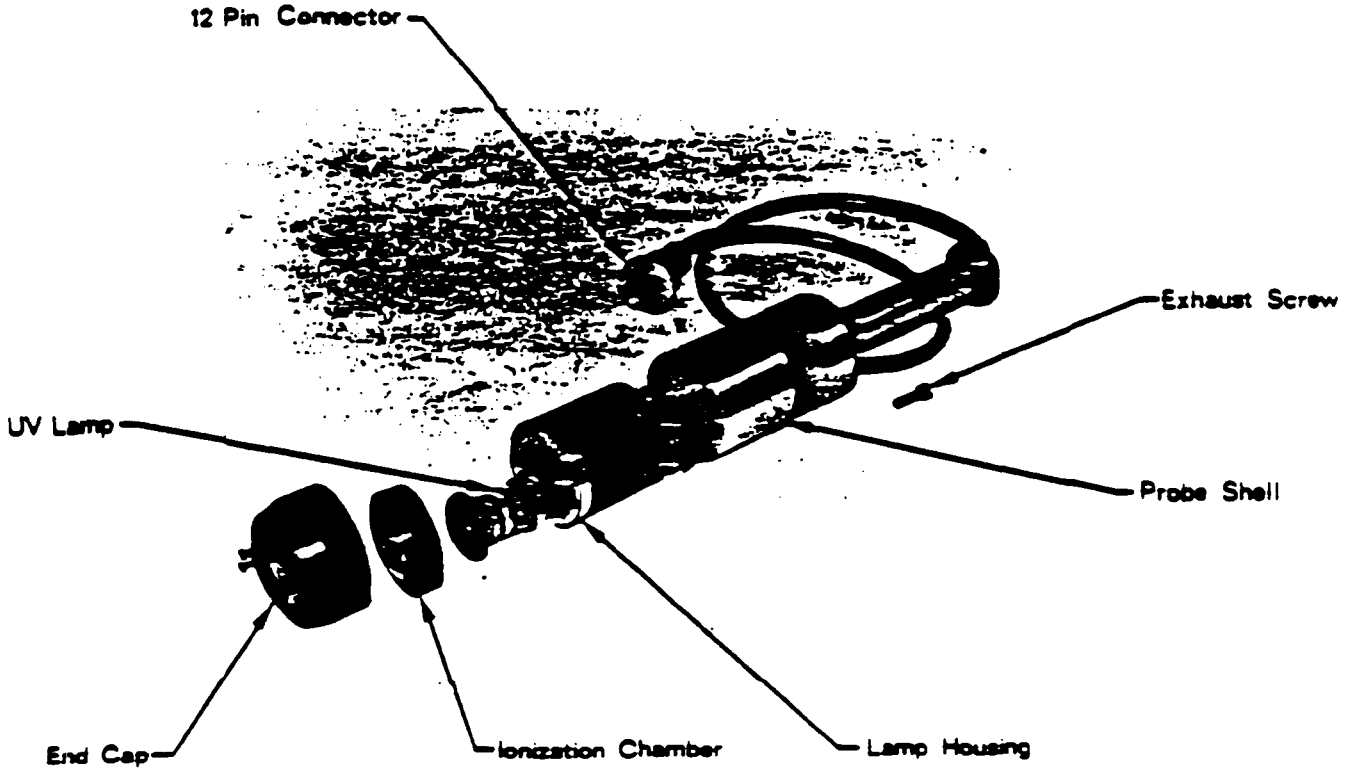


FIGURE 3 COMPONENT PARTS OF PROBE

4.1.3 Instrument appears to be functional, but responses are lower than expected or erratic. The window of the light source may be dirty and need to be cleaned. To clean the light source window:

1. Disassemble the probe assembly by repeating steps 1 through 6 under 4.1.2 above.
2. Clean the window of the light source using compound provided with instrument and soft clean cloth. Important: use cleaning compound on the window of the 10.2 eV lamp only. The cleaning compound may damage the windows of the 9.5 and 11.7 eV lamps.
3. Reassemble the probe assembly repeating step 7 through 12 above.

4.2 Specific Faults

4.2.1 No meter response in any switch position (including BATT CHK)

1. Broken meter movement: Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.
2. Electrical connection to meter is broken: Check all wires leading to meter and clean the contacts of quick-disconnects.
3. Battery is completely dead: Disconnect battery and check voltage with a volt-ohm meter.
4. Check 2 amp fuse.
5. If none of the above solves the problem, consult the factory.

4.2.2 Meter responds in BATT CHK position, but reads zero or near zero for all others.

1. Power supply defective: Check power supply voltages per Figure 4. If any voltage is out of specification, consult the factory.

2. Input transistor or amplifier has failed: Rotate zero control; meter should deflect up/down as control is turned. Open probe; both transistors should be fully seated in sockets.

3. Input signal connection broken in probe or readout: Check input connector on printed circuit board. Should be firmly pressed down. Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object. Check all wires in readout for solid connections.

4.2.3 Instrument responds correctly in BATT CHK, and STBY, but not in measuring mode.

1. Check to see the light source is on (See Section 4.1.2).

2. Check high voltage power supply (see Figure 4).

3. Open end of probe, remove lamp and check high voltage on lamp contact ring.

4. If high voltage is present at all above points, light source has most likely failed. Consult the factory.

4.2.4 Instrument responds correctly in all positions, but signal is lower than expected.

1. Check span setting for correct value.

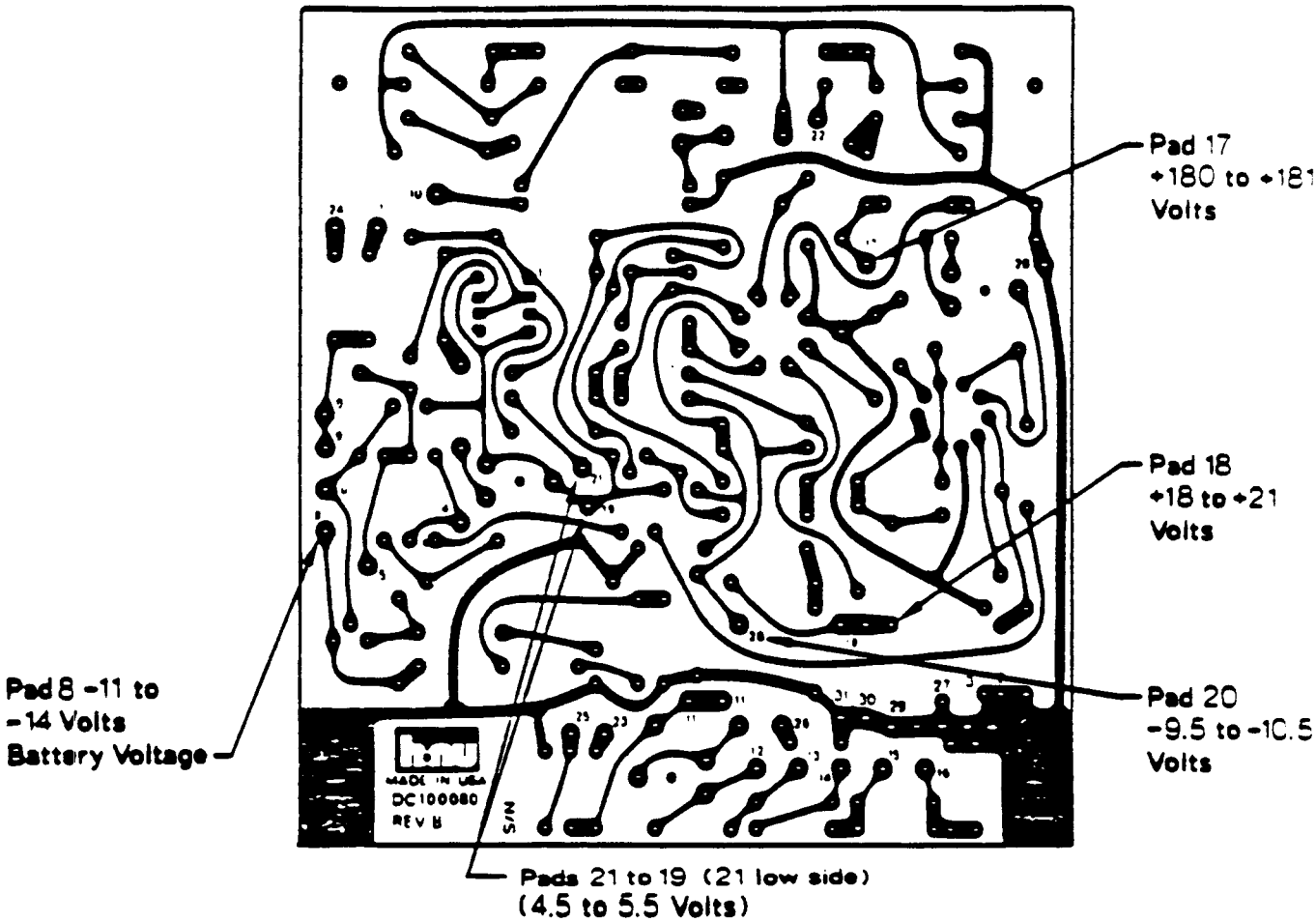
2. Clean window of light source (See 4.1.3).

3. Double check preparation of standards.

4. Check power supply 180 V output. See Figure 4.

5. Check for proper fan operation. Check fan voltage. See Figure 4.

6. Rotate span setting. Response should change if span pot is working properly.



All Voltages Respect to Ground							
pads	voltage	pads	voltage	pads	voltage	pads	voltage
1	− 5.7 V	9	− 12.2V	17	180V	25	0
2	GRD	10	− 12.1V	18	+ 19.4V	26	0
3	GRD	11	0	19	− 10.6V	27	GRD
4	−10 7V	12	0	20	− 9.7V	28	0
5	− 11.3V	13	0	21	− 14.5V	29	GRD
6	− 12.1V	14	0	22	− 400V	30	GRD
7	0	15	0	23	0	31	GRD
8	− 12.2V	16	0	24	0		

Figure 4 Power Supply PC Board

4.2.5 Instrument responds in all switch positions, but is noisy (erratic meter movement).

1. Open circuit in feedback circuit. Consult the factory.
2. Open circuit in cable shield or probe shield. Consult the factory.

4.2.6 Instrument response is slow and/or irreproducible.

1. Fan operating improperly. Check fan voltage. See Figure 4.
2. Check calibration and operation.

4.2.7 Low battery indicator.

1. Indicator comes on if battery charge is low.
2. Indicator also comes on if ionization voltage is too high.

CALIBRATION AND MAINTENANCE PROCEDURES
CENTURY SYSTEMS PORTABLE ORGANIC VAPOR ANALYZER MODEL OVA-128

1.0 INTRODUCTION

This procedure presents steps required to calibrate and maintain the model OVA-128 organic vapor analyzer. Specifications and operating principles and procedures are presented in Procedure 5607003.

2.0 CALIBRATION

2.1 General

The OVA is capable of responding to nearly all organic compounds. However, the response will vary from compound to compound. The responses of some compounds relative to methane, are presented in Table 1. For precise analyses it is necessary to calibrate the instrument to a specific compound of interest, particularly if that compound contains elements other than carbon and hydrogen. For general use, the instrument is calibrated to methane.

Internal electronic adjustments are provided to calibrate and align the electronic circuits. There are four such adjustments, all located on the electronics board. One adjustment potentiometer, R-38, is factory set and is used to set the power supply voltage. Potentiometer R-38 thus should never be adjusted. The remaining three adjustments, R-31 (X1), R-32 (X10), and R-33 (X100) are used for setting the electronic amplifier gain for each of the three calibration ranges. Access to the adjustments is accomplished by removing the instrument from its case.

TABLE 1 - RESPONSE OF OVA TO HYDROCARBONS RELATIVE TO METHANE

Compound	Relative Response (%)
Methane	100 (Reference)
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Tolvene	120
Ethane	90
Acetone	60
Methyl Ethyl Ketone	80
Methyl Isobutyl Ketone	100
Methyl Alcohol	15
Ethyl Alcohol	25
Isopropyl Alcohol	65
Carbon Tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

2.2 Methane Calibration

2.2.1 Equipment

- o Calibration gas (100 ppm methane)
- o T-tube assembly

2.2.2 Instrument Startup

Start instrument by:

1. Move PUMP switch to ON and check battery condition by moving the INSTR switch to the BATT position.

Recharge battery (section 3.0) if battery level is low.

2. Move INSTR switch to ON and allow 5 minutes for warmup.

3. Place instrument in vertical position and check flow rate.

4. Open the H_2 TANK VALVE and H_2 SUPPLY VALVE.

5. Depress Ignitor button for 6 seconds or until hydrogen ignites, whichever is shorter. If hydrogen fails to ignite, wait 2 minutes and attempt to ignite hydrogen.

6. Once lit, wait 5 minutes for instrument to stabilize before starting calibration procedure.

7. Open instrument cover to expose circuit board.

2.2.3 Calibration

Calibration should be performed in a well ventilated area.

1. Set CALIBRATE switch to X10.

2. For methane calibration the GAS SELECT control should be set to 300. Check to ensure that this control is set at 300.

3. Adjust meter reading to zero by rotating the Calibrate ADJUST (zero) knob.

4. Attach one end of T assembly to methane gas cylinder and the other to the probe.

5. Crack open methane gas cylinder until a slight flow of gas can be detected exiting the open end of the T assembly.

6. Adjust trimpot R-32 on circuit board so that meter reads the equivalent of the calibration gas concentration. This sets the instrument gain for methane with the panel mounted gain adjustment set at 300.

7. Close methane gas cylinder. Turn off H_2 SUPPLY VALVE to put out flame. Wait for flameout alarm to sound to ensure the flame is out.

8. Leave CALIBRATE switch on the X10 position and use CALIBRATE ADJUST (zero) knob to adjust meter reading to 4 ppm.

9. Place CALIBRATE switch in X1 position and, using trimpot R-31 on circuit board, adjust meter reading to 4 ppm. This is the Bias Adjustment for the X1 range.

10. Move CALIBRATE switch to X10 position again. Use CALIBRATE ADJUST (zero) knob to adjust meter to a reading of 40 ppm.

11. Move CALIBRATE switch to X100 position and use trimpot R-33 on circuit board to adjust meter reading to 40 ppm.

12. Move CALIBRATE switch to X10 position and use CALIBRATE ADJUST (zero) knob to adjust meter reading to zero.

13. Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.

14. Shut instrument down by ensuring that the H_2 SUPPLY VALVE and H_2 TANK VALVE are closed and the INSTR and PUMP switches are in the OFF position.

15. Record on instrument calibration label, calibration date, gas, and initials of person performing calibration. Remove old tag and replace it with updated label. Fill out instrument history log form.

2.3 Calibration to Specialty Gas/Vapor

Primary calibration of the instrument is accomplished using a known mixture of a specific gas or vapor.

2.3.1 Equipment

- Calibration (span) gas (75-90ppm of known gas or vapor)
- T-tube assembly

2.3.2 Instrument Startup

Follow steps in 2.2.2 above.

2.3.3 Calibration

Calibration should be performed in a well ventillated area.

1. Set CALIBRATE switch to 10.
2. Adjust meter reading to zero by rotating the CALIBRATE ADJUST (zero) knob.
3. Attach one end of T assembly to calibration gas cylinder and the other to the probe.
4. Crack open calibration gas cylinder until a slight flow of gas can be detected exiting the open end of the T assembly. (Caution: if the calibration gas is toxic or highly flammable, calibration should occur underneath a hood.)
5. Adjust GAS SELECT knob on instrument until the meter reads the same level as that of the calibration gas.
6. Turn off calibration cylinder and remove T assembly.
7. The instrument is now calibrated for the specialty gas/vapor. All responses of the instrument should be recorded relative to the specialty gas.
8. Calibration in the X10 range by adjusting the GAS Select knob automatically calibrates the instrument for the X1 and X100 ranges. No further adjustments are necesary.
9. Shut instrument down by closing the H_2 SUPPLY VALVE and H_2 TANK VALVE, and putting the INSTR and PUMP switches in the OFF position.
10. Record in instrument calibration label calibration date, span gas and concentration, span setting, and initials of person performing calibration. Remove old

tag and replace it with updated label. Fill out instrument history log.

3.0 FILLING OF HYDROGEN SUPPLY

The instrument should be completely shut down for hydrogen tank refilling. The refilling should be done in a ventillated area. There should be no potential ignitors or flame in the area.

1. Attach filling hose to external source of hydrogen. Pure hydrogen of 1,600 to 2,300 psi will be required.
2. Crack open valve on hydrogen supply, place FILL/BLEED valve on hose in FILL position and purge hose for 2-3 seconds.
3. Close FILL/BLEED Valve (OFF position) and immediately attach other end of fill hose to instrument fill connection and tighten the connection.
4. Open supply valve external on hydrogen tank 1/2 to 1 turn. Set regulator to 1,600 to 2,300 psi.
5. Open the REFILL Valve and the H_2 Tank VALVE on the instrument.
6. Place FILL/BLEED Valve in FILL position. The instrument hydrogen tank should now be filling.
7. The instrument H_2 Pressure Indicator should now indicate instrument tank pressure. This pressure should approximate that of the external supply tank regulator gauge.
8. After the instrument tank is filled, shut off:
   a. The REFILL VALVE on the instrument panel.
   b. The FILL/BLEED Valve on the filling hose assembly.
   c. The valve on the external hydrogen supply bottle.
9. The supply hose and internal lines on the instrument now contain hydrogen under pressure. To reduce this pressure to atmospheric pressure:
   a. Turn FILL/BLEED Valve to BLEED position until gas can no longer be heard escaping.

b. Turn FILL/BLEED Valve to FILL position to allow gas trapped in the connective fittings to go into the hose assembly.

c. Turn FILL/BLEED Valve to BLEED position to bleed off this pressure.

d. Turn FILL/BLEED Valve to OFF position. Keep valve in OFF position.

10. Close H_2 Tank Valve.

11. Remove fill hose from instrument. Any gas still under pressure will escape as the connector is removed. Release of gas should only be momentary.

12. As a check of the integrity of the instrument's hydrogen supply system, observe the H_2 TANK PRESSURE Indicator with the system shut down. Release of internal pressure should not go down rapidly. If there is a rapid decrease (greater than 350 PSIG/hour) with the instrument in shut down mode, there is a significant leak in the H_2 supply system. If so, the instrument should be returned to the manufacturer for repairs.

4.0 BATTERY RECHARGING

The battery should be recharged following each use of 4 hours or more, or when the battery check indicator indicates need of a charge. Never charge instrument in hazardous environment or when refilling hydrogen tank.

1. Remove cover from battery charge part on instrument.

2. Plug charger BNC connector into mating connector on battery cover and insert AC plug into 115 VAC wall outlet.

3. Move battery charger switch to the ON position. The light above the switch should illuminate.

4. Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the right while charging. When fully charged, the pointer will be in line with the charged mark above the scale.

5. Approximately 1 hour of charging time is required for each hour of operation; 12 hours for complete charge. Do not charge for more than 24 hours.

5.0 MAINTENANCE

5.1 General

Section 6.0 of the Model OVA-128 Instruction and Service Manual contains detailed maintenance and repair procedures for servicing the OVA. These procedures are not repeated here. Equipment managers are referred to the manual for repair of the OVA.

Equipment managers should be thoroughly familiar with instrument operation before performing maintenance. It is essential that all safety considerations regarding use and maintenance of this instrument be understood. There should be no potential igniters or flame in the area when filling, emptying, or purging the hydrogen system and the instrument should be in shut-down mode.

5.2 Trouble Shooting

Table 2 presents common problems and corrective actions for repairing the instrument.

6.0 REFERENCE

Foxboro Analytical. No date. Instruction and Service Manual, Century Systems Portable Organic Vapor Analyzer Model OVA-128 (M1 2R900AC).

TABLE 2

TROUBLE	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator	a) Check teflon tubing on valve assembly for kinks, etc.	Straighten or replace teflon tubing
	b) Check flow rate with valve in down position.	Check for over restriction of charcoal filter
2) H2 flame will not light	a) Check column connections on top of unit to make sure they are tight.	Tighten fittings
	b) Check column for sharp bends or kinks. (Hydrogen flows through this column at all times and a sharp bend will compact packing too tightly for proper hydrogen flow.)	Replace column
	c) Check charcoal filter fittings to make sure they are tight.	Tighten fittings
	d) Check H2 flow rate from the column.	Adjust hydrogen pressure to obtain 12 cc/min. flow rate.
	e) Check that the Inject and Backflush Valves are both completely in or out. A partially activated valve will block the H2 and air flow paths.	Ensure both valves are either completely in or out.
	f) If a new column was installed prior to problem identification, check for proper hydrogen flow rate through the column (should be approximately 12 cc/minute). Reference paragraph 7.1.4.2 d.	Increase hydrogen pressure to obtain proper hydrogen flow rate or if column is excessively restrictive, replace or repack the column.
3) Ambient background reading in clean environment is too high	a) Check for contamination in charcoal filter assembly. This can be detected if ambient reading increases when going in to the chromatographic mode. Reference paragraph 7.1.5.2 b.	Replace activated charcoal in charcoal filter assembly.
	b) Check for contamination in column. Reference paragraph 7.1.5.2 a	Replace or clean column.
	c) Check for contamination in column valve assembly.	Remove valve stems and wipe with clean lint-free cloth. Heat valve assembly during operation to vaporize and remove contaminants.
4) Flame-out when operating either valve	a) Ensure valves are being operated with a quick, positive motion.	Operate valve with a positive motion.
	b) Either H2 or air may be leaking around one or more of the valve quad rings. Assess by taste and "O" ring inspection.	Remove stems and lightly coat with silicone grease, only on contact surface of the "O" ring. Wipe off excess (do not remove quad rings).
	c) Damaged or worn quad rings causing leak.	Replace quad rings and grease as above.
5) Excessive peak tailing	a) Change or clean GC column and see if problem disappears.	Ensure columns are clean prior to use. Refer to paragraph 7.1.5.2 a for cleaning instructions. If one of a same type of column tails worse than others, repack the column or discard.
	b) Inspect GC valves for excessive silicone grease or contamination.	Excessive lubricant or foreign matter in the valve assembly can cause excessive tailing. Clean valve assemblies and lightly relubricate as required. Lubricant should be put only on the outside contact surface of the "O" ring. Do not get grease into "O" ring

APPENDIX D

IEPA SAMPLE BOTTLE SUPPLY SERVICE

SAMPLE CONTAINER AND COMPONENT MATERIAL SPECIFICATIONS

Figure B-1 following, designates the specifications for the eight types of containers and the associated materials (i.e., teflon liners, lids, etc.) to be supplied by the Contractor under this contract.

All materials received from vendors shall be subjected to incoming inspection by the Contractor to insure conformance with these established specifications. Variations in materials shall be considered unacceptable. Any materials not in conformance with these specifications shall be returned by the Contractor to the vendor for replacement.

Note: CDM will request that bottles be cleaned for drinking water VOA analysis. The procedures to be used for this container preparation and cleaning process are presented in Exhibit A, Section C, Part II, beginning on the third page following.

FIGURE B-1

Container Type	Container and Material Specification	Parameter and Sample Type
	Container: 1 liter* amber, Boston round, glass bottle, 33 mm pour-out neck finish Closure: white polypropylene cap, 33-400 size, .015 mm teflon liner	Extractable Organics
3	Container: 1 liter* natural high-density polyethylene cylinder round bottle, 52g weight, 28 mm neck finish. Closure: baked polyethylene, white ribbed, 28-400 or 28-410 size; unlined lid.	Metals, Cyanide Radioactivity, General, Nutrients, Sulfide
5	Container: 32 oz. tall, wide-mouth straight-sided paragon, flint glass jar, 89 mm neck finish. Closure: white polypropylene cap, 89-400 size, .015 mm teflon liner	Extractable Organics, Oil/ Grease, Metals, Mercury, Cyanide, Nutrients, Phenols, General, Sulfide
7	Container: 8 oz. wide-mouth glass jar	Same as type 5
8	Container: 40 ml borosilicate glass vial, Type 1 glass, 24 mm neck finish. Closure: black phenolic, open-top, screw cap, 15 cm opening, 24-400 size. Septum: 22 mm disc of 2 mil teflon bonded to silicon for total thickness of 125 mil.	THM/VOA
9	Container: 1/2 gallon amber glass, ring handle bottle/jug, 38 mm neck finish. Closure: teflon-lined white propylene cap, 38-400 size.	Extractable Organics

10	Container: 500 ml natural high density polyethylene, oblong bottle, 43 mm neck finish. Closure: white propylene unlined cap, 43-400 size (or 43 mm).	Mercury
11	1 gallon plastic	Prefiltration
12	Container: 2 oz., wide-mouth straight-sided paragon, flint glass jar, 53 mm neck closure: white polypropylene cap, 53-400 size. 0.015 mm teflon liner.	THM/VOA

* These bottles must have sufficient overfill to accommodate an actual capacity of 1 liter of liquid. Bottle manufacturers refer to these bottles as 32 ounce bottles, however all 32 ounce bottles do not have sufficient overfill to meet the requirement.

NOTE: Containers and component material specifications different than, but equivalent to, the manufacturer's specifications cited herein may be acceptable. The bidder shall be required to demonstrate equivalence prior to Government approval of use of alternate materials. The Government shall determine acceptability as part of bidder preaward confirmations (see Pre-Award Bid Confirmations).

CONTAINER PREPARATION AND CLEANING PROCEDURES

The Contractor shall clean and prepare containers and component materials according to the following procedures specified for each container type.

I. Extractable Organics

Container Types:
1 - 1 liter amber glass
5 - 32 oz glass jar
9 - 1/2 gallon amber glass
7 - 8 oz glass jar

1. The containers, teflon liners and caps are to be washed in hot tap water with laboratory-grade non-phosphate detergent.
2. Rinse three times with tap water.
3. Rinse three times with ASTM Type I organic-free water.
4. Dry in oven @ 125°C for one hour.
5. Rinse inside and outside of container with pesticide hexane.
6. Dry containers, liners, and caps in an oven at 125°C for one hour.
7. Allow containers to cool and seal with teflon lined caps.
8. Label each container with color coded labels, with lot number, and pack in a sealable carton.
9. Place identical labels on exterior of carton and store in a designated contaminant-free area.

II. Purgeable Organics:

Container Types:
8 - 40 ml glass vial
12 - 2 oz. glass jar

1. Containers, teflon-backed septa and caps are washed in hot tap water with laboratory-grade non-phosphate detergent.
2. Rinse three times with tap water.
3. Rinse three times with ASTM Type I organic-free water.
4. Oven dry vials, containers, caps, septa, and teflon-lined lids at 125°C for one hour.

5. Cool in a contaminant-free area.

6. Seal vials with septa (teflon side down) and cap. Seal containers with cap and liner.

7. Label each vial and container with color coded label with lot number, and pack in a carton and seal.

8. Place identical label on outside of carton with respective lot number and store in a contaminant-free area.

III. Metals, Mercury, Cyanide, Radioactivity

Container Types: 3 - 1L high-density Polyethylene
5 - 32 oz glass jar 10 - 250 ml
high-density Polyethylene 7 - 8 oz glass jar

1. The bottles and caps are washed in tap water with laboratory grade non-phosphate detergent.

2. Rinse with 50% reagent grade HN03.

3. Rinse three times with ASTM Type I deionized water.

4. Invert and dry in a contaminant-free area.

5. Cap each container, label with color coded label with lot number and place in a carton.

6. Label carton with the same lot number and store in a contaminant-free area.

IV. Phenols, Nutrients, General, Prefiltration, Sulfide

Container Types: 3 - IL high-density Polyethylene
5 - 32 oz glass jar
11 - 1 gallon plastic
7 - 8 oz glass jar

1. Wash containers in tap water with laboratory-grade non-phosphate detergent. Wash caps in a separate wash.

2. Rinse three times with tap water.

3. Rinse three times with ASTM Type I deionized water.

4. Invert bottles and dry in a contaminant-free area.

3. Cap bottles and label with color coded label with lot number and pack a carton.

5. Label the carton with the same lot number and store in a contaminant-free area.

/ Oil and Grease

Container Types: 5 - 32 oz glass jar
7 - 8 oz glass jar

1. The containers, teflon liners, and caps are washed in hot tap water with laboratory-grade non-phosphate detergent.

2. Rinse three times with tap water.

3. Rinse with ASTM Type I deionized water.

4. Dry in oven at 105°C for one hour.

5. Allow containers to cool and seal with teflon lined caps.

6. Label each container with color coded labels with lot number and pack in a sealable carton.

7. Place identical labels on exterior of carton and store in a designated contaminant-free area.

APPENDIX E

QA/QC MANUAL AND ANALYTICAL METHODS - FAST-TURNAROUND LABORATORY

METHOD 8010

METHOD 8010

HALOGENATED VOLATILE ORGANICS

1.0 SCOPE AND APPLICATION

1.1 Method 8010 is used to determine the concentration of various volatile halogenated organic compounds. Table 1 indicates compounds that may be analyzed by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit for other matrices.

See Addendum I

2.0 SUMMARY OF METHOD

2.1 Method 8010 provides gas chromatographic conditions for the detection of halogenated volatile organic compounds. Samples can be analyzed using direct injection or purge-and-trap (Method 5030). Ground water samples must be analyzed using Method 5030. A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a halogen-specific detector (HSD).

See Addendum I

3.0 INTERFERENCES

3.1 Refer to Methods 5030 and Addendum II

3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas Chromatograph: analytical system complete with gas chromatograph suitable for on-column injections or purge-and-trap sample introduction and all required accessories, including detector, analytical columns, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

See Addendum I

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS FOR HALOGENATED VOLATILE ORGANICS

Compound	Retention time (min) Col. 1	Retention time (min) Col. 2	Method detection limit[a] (ug/L)
Benzyl chloride			
Bis(2-chloroethoxy)methane			
Bis(2-chloroisopropyl)ether			
Bromobenzene			
Bromodichloromethane	13.7	14.6	0.10
Bromoform	19.2	19.2	0.20
Bromomethane			
Carbon tetrachloride	13.0	14.4	0.12
Chloroacetaldehyde			
Chlorobenzene	24.2	18.8	0.25
Chloroethane	3.33	8.68	0.52
Chloroform	10.7	12.1	0.05
1-Chlorohexane			
2-Chloroethyl vinyl ether	18.0		0.13
Chloromethane	1.50	5.28	0.08
Chloromethylmethyl ether			
Chlorotoluene			
Dibromochloromethane	16.5	16.6	0.09
Dibromomethane			
1,2-Dichlorobenzene	34.9	23.5	0.15
1,3-Dichlorobenzene	34.0	22.4	0.32
1,4-Dichlorobenzene	35.4	22.3	0.24
Dichlorodifluoromethane			
1,1-Dichloroethane	9.30	12.6	0.07
1,2-Dichloroethane	11.4	15.4	0.03
1,1-Dichloroethylene	8.0	7.72	0.13
trans-1,2-Dichloroethylene	10.1	9.38	0.10
Dichloromethane			
1,2-Dichloropropane	14.9	16.6	0.04
trans-1,3-Dichloropropylene	15.2	16.6	0.34
1,1,2,2-Tetrachloroethane	21.6		0.03
1,1,1,2-Tetrachloroethane			
Tetrachloroethylene	21.7	15.0	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
1,1,2-Trichloroethane	16.5	18.1	0.02
Trichloroethylene	15.8	13.1	0.12
Trichlorofluoromethane	7.18		
Trichloropropane			
Vinyl chloride	2.67	5.28	0.18

[a] Using purge-and-trap method (Method 5030).
Refer to Addendum 2 for updated Table 1.

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-level soil	10
Water miscible liquid waste	500
High-level soil and sludge	1250
Non-water miscible waste	1250

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

4.1.2 **Columns:**

4.1.2.1 Column 1: 8-ft x 0.1-in I.D. stainless steel or glass column packed with 1% SP-1000 on Carbopack-B 60/80 mesh or equivalent.

4.1.2.2 Column 2: 6-ft x 0.1-in I.D. stainless steel or glass column packed with chemically bonded n-octane on Porasil-C 100/120 mesh (Durapak) or equivalent.

4.1.3 **Detector:** Electrolytic conductivity (HSD).

4.2 Sample introduction apparatus: Refer to Method 5030 for the appropriate equipment for sample introduction purposes.

4.3 Syringes: 5-mL Luerlok glass hypodermic and a 5-mL, gas-tight with shutoff valve.

4.4 Volumetric flask: 10-, 50-, 100-, 500-, and 1,000-mL with a ground-glass stopper.

4.5 Microsyringe: 10-, 25-uL with a 0.006-in I.D. needle (Hamilton 702N or equivalent) and a 100-uL.

5.0 REAGENTS

5.1 Reagent water: See Addendum II

5.2 Stock standards: Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standards in methanol using assayed liquids or gases, as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials of the toxicity of these materials should be prepared in a hood. See Addendum I

5.2.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.2.2 Add the assayed reference material, as described below.

5.2.2.1 Liquids: Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.2.2.2 Gases: To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol

meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas rapidly dissolves in the methanol. This may also be accomplished by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side-arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.2.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.2.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10°C to -20°C and protect from light.

5.2.5 Prepare fresh standards every 2 months for gases or for reactive compounds such as 2-chloroethylvinyl ether. All other standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.3 Secondary dilution standards: Using stock standard solutions, prepare in methanol secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 5.4 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. See Addendum II

5.4 Calibration standards: Calibration standards at a minimum of five concentration levels are prepared in reagent water from the secondary dilution of the stock standards. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for detection by this method (e.g., some or all of the compounds listed in Table 1 may be included). In order to prepare accurate aqueous standard solutions, the following precautions must be observed. See Addendum II

5.4.1 Do not inject more than 20 uL of alcoholic standards into 100 mL of reagent water.

5.4.2 Use a 25-uL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

5.4.3 Rapidly inject the alcoholic standard into the filled volumetric flask. Remove the needle as fast as possible after injection.

5.4.4 Mix aqueous standards by inverting the flask three times only.

5.4.5 Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use any solution contained in the neck of the flask).

5.4.6 Never use pipets to dilute or transfer samples or aqueous standards.

5.4.7 Aqueous standards are not stable and should be discarded after 1 hr, unless properly sealed and stored. The aqueous standards can be stored up to 24 hr, if held in sealed vials with zero headspace. See Addendum II

5.5 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes (Paragraph 5.6) have been used successfully as internal standards, because of their generally unique retention times. See Addendum II

5.5.1 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest as described in Section 5.4.

5.5.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 5.2 and 5.3. It is recommended that the secondary dilution standard be prepared at a concentration of 15 ug/mL of each internal standard compound. The addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 ug/L.

5.5.3 Analyze each calibration standard according to Section 7.0, adding 10 uL of internal standard spiking solution directly to the syringe.

5.6 Surrogate standards: The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 5.2, add a volume to give 750 ug of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix, and dilute to volume for a concentration of 15 ng/uL. Add 10 uL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. If the internal standard calibration procedure is used, the surrogate compounds may be added directly to the internal standard spiking solution (Paragraph 5.5.2). See Addendum I

5.7 Methanol: pesticide quality or equivalent. Store away from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See Addenda I, II

7.0 PROCEDURE

7.1 Volatile compounds are introduced into the gas chromatograph either by direct injection or purge-and-trap (Method 5030). Method 5030 may be used directly on ground water samples or low-level contaminated soils and sediments. For medium-level soils or sediments, methanolic extraction, as described in Method 5030, may be necessary prior to purge-and-trap analysis.

7.2 Gas chromatography conditions (Recommended):

See Addendum II

7.2.2 Column 2: Set helium gas flow at 40 mL/min flow rate. Set column temperature at 50°C for 3 min; then program a 6°C/min temperature rise to 170°C and hold for 4 min.

7.3 Calibration:

See Addendum II

7.3.1 Calibration must take place using the same sample introduction method that will be used to analyze actual samples (see Paragraph 7.4.1).

7.3.2 The procedure for internal or external calibration may be used.

7.4 Gas chromatographic analysis: See Addenda I, II

7.4.1 Introduce volatile compounds into the gas chromatograph using either Method 5030 (purge-and-trap method) or the direct injection method (see Paragraph 7.4.1.1). If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to purging.

7.4.1.1 Direct injection: In very limited applications (e.g., aqueous process wastes) direct injection of the sample into the GC system with a 10-uL syringe may be appropriate. The detection limit is very high (approximately 10,000 ug/L) therefore, it is only

permitted where concentrations in excess of 10,000 ug/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

7.4.2 Follow Section 7.4 in Method 8240 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.3 See Addendum II

7.4.4 Record the sample volume purged or injected and the resulting peak sizes (in area units or peak heights).

7.4.5 See Addendum I

7.4.6 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second GC column is recommended.

7.4.7 If the response for a peak is off-scale, prepare a dilution of the sample with reagent water. The dilution must be performed on a second aliquot of the sample which has been properly sealed and stored prior to use.

8.0 QUALITY CONTROL See Addendum II

8.1

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ADDENDUM I

ADDENDUM I

1.0 SCOPE AND APPLICATION

1.1 Table 2 lists compounds and acceptable detection limits.

2.0 SUMMARY OF METHOD

2.1 All samples will be analyzed using Method 5030. See attached Method 5030.

2.2 Delete section.

3.0 INTERFERENCES

3.1 Refer to Methods 5030 and 8240.

4.0 APPARATUS AND MATERIALS

4.1.1 Gas Chromatograph with Mass Spectrometer will be used as the detector. The instrument is an ion-trap Mass Spectrometer with a Compag 386E data system for measuring peak heights and areas.

5.0 REAGENTS

5.1 Reagent Water: See Table 1 Monitoring Frequency.

5.2 Stock Standards: Stock solutions are purchased as certified solutions.

5.3 Secondary Dilution Standards: The secondary dilution standards will be prepared at concentrations of .4ul/ml to 20ul/ml and will be stored with no headspace for volatiles. See Table 2.

5.6 Surrogate Standards: The analyst will monitor both the performance of the analytical system and effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate. The surrogate combinations will be 4 - Bromofluorobenzene, dibromofluoromethane and Toluene-d8 ranging from 0.4ul/ml to 20ul/ml. The internal standard calibration procedure will be used and the surrogate compounds will be added directly to the internal standard spiking solution.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See Page III; all samples are then placed in a secure refrigerator until sample analysis starts.

7.0 PROCEDURE

7.2 Gas Chromatography conditions:

7.2.1 Column 1 - DB-624: Set helium gas flow at 40 ml/min flow rate. Set column temperature at 45°C for 3 min., then program an 8°C/min temperature rise to 220° and hold for 15 min.

7.3 Calibration: See Method 8240.

7.3.1 Calibration will take place using same sample introduction Method 5030 Trap and Purge, that will be used to analyze actual sample.

7.3.2 The procedure for external calibration will be used. It will be Bromofluorobenzene tuning calibration.

7.4 Introduce volatile compounds into gas chromatograph using Method 5030 and add 4ul of internal standard to the sample prior to purging.

7.4.5 See Method 8240.

8.0 QUALITY CONTROL

See Method 8240 plus forms 1 - 5.

All information is included in Method 8240.

TABLE 1

COMPOUND	RETENTION TIME (MINUTES)
Ethylene oxide	1.30
Chloromethane	2.30
Dichlorodifluoromethane	2.47
Bromomethane	3.10
Vinyl chloride	3.80
Acetonitrile	3.97
Chloroethane	4.60
Methyl iodide	5.37
Methylene chloride	6.40
Carbon disulfide	7.47
Trichlorofluoromethane	8.30
Propionitrile	8.53
Allyl chloride	8.83
1,1-Dichloroethene	9.00
Bromochloromethane (I.S.)	9.30
Allyl alcohol	9.77
trans-1,2-Dichloroethene	10.00
1,2-Dichloroethane	10.10
Propargyl alcohol	10.77
Chloroform	11.40
1,2-Dichloroethane-d_4(surr)	12.10
2-Butanone	12.20
Methacrylonitrile	12.37
Dibromomethane	12.53
2-Chloroethanol	12.93
b-Propiolactone	13.00
Epichlorohydrin	13.10
1,1,1-Trichloroethane	13.40
Carbon tetrachloride	13.70
1,4-Dioxane	13.70
Isobutyl alcohol	13.80
Bromodichloromethane	14.30
Chloroprene	14.77
1,2:3,4-Diepoxybutane	14.87
1,2-Dichloropropane	15.70
cis-1,3-Dichloropropene	15.90
Bromoacetone	16.33
Trichloroethene	16.50
Benzene	17.00
trans-1,3-Dichloropropene	17.20
1,1,2-Trichloroethane	17.20
3-Chloropropionitrile	17.37
1,2-Dibromoethane	18.40
Pyridine	18.57
2-Chloroethyl vinyl ether	18.60
2-Hydroxypropionitrile	18.97
1,4-Difluorobenzene (I.S.)	19.60
Malononitrile	19.60
Methylmethacrylate	19.77
Bromoform	19.80

TABLE 1

COMPOUND	RETENTION TIME (MINUTES)
1,1,1,2-Tetrachloroethane	20.33
1,3-Dichloro-2-propanol	21.83
1,1,2,2-Tetrachloroethane	22.10
Tetrachloroethene	22.20
1,2,3-Trichloropropane	22.20
1,4-Dichloro-2-butene	22.73
n-Propylamine	23.00
2-Picoline	23.20
Toluene	23.50
Ethyl methacrylate	23.53
Chlorobenzene	24.60
Pentachloroethane[a]	24.83
Ethylbenzene	26.40
1,2-Dibromo-3-chloropropane	27.23
4-Bromofluorobenzene (surr.)	28.30
Benzyl chloride	29.50
Styrene	30.83
Acetone	1.21
Acrolein	1.56
Acrylonitrile	2.12
Chlorobenzene-d_5 (I.S.)	13.50
Chlorodibromomethane	20.50
1,1-Dichloroethane	12.01
Ethanol	9.40
2-Hexanone	16.00
Iodomethane	23.05
4-Methyl-2-pentanone	19.15
Toluene-d_8 (surr.)	22.17
Vinyl acetate	1.04
Xylene (Total)	16.07

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TABLE 2

	CAS No.	ADL	RESULTS
Benzene	71-43-2	0.002	
Bromobenzene	108-86-1	0.002	
Bromochloromethane	74-97-5	0.002	
Bromodichloromethane	75-27-4	0.001	
Bromoform	75-25-2	0.002	
Bromomethane	74-83-9	0.002	
n-Butylbenzene	104-51-8	0.002	
sec-Butylbenzene	135-98-8	0.002	
tert-Butylbenzene	98-06-6	0.002	
Carbon tetrachloride	56-23-5	0.002	
Chlorobenzene	108-90-7	0.002	
Chloroethane	75-00-3	0.002	
Chloroform	67-66-3	0.0005	
Chloromethane	74-87-3	0.002	
2-Chlorotoluene	95-49-8	0.002	
4-Chlorotoluene	106-43-4	0.002	
Dibromochloromethane	124-38-1	0.002	
1,2-Dibromo-3-chloropropane	96-12-8	0.002	
1,2-Dibromoethane	106-93-4	0.002	
Dibromomethane	74-95-3	0.002	
1,2-Dichlorobenzene	95-50-1	0.002	
1,3-Dichlorobenzene	541-73-1	0.002	
1,4-Dichlorobenzene	106-46-7	0.002	
Dichlorodifluoromethane	75-71-8	0.002	
1,1-Dichloroethane	75-34-3	0.0007	
1,2-Dichloroethane	107-06-2	0.0003	
1,1-Dichloroethene	75-35-4	0.0013	
cis-1,2-Dichloroethene	156-69-4	0.005	
trans-1,2-Dichloroethene	156-60-5	0.001	
1,2-Dichloropropane	78-87-5	0.002	
1,3-Dichloropropane	142-28-9	0.002	
2,2-Dichloropropane	594-20-7	0.002	
1,1-Dichloropropene	563-58-6	0.002	
Ethylbenzene	100-41-1	0.002	
Hexachlorobutadiene	87-68-3	0.002	
Isopropylbenzene	98-82-8	0.002	
p-Isopropyltoluene	99-87-6	0.002	

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	CAS No.	ADL	RESULTS
Methylene chloride	75-09-2	0.005	
Naphthalene	91-20-3	0.002	
n-Propylbenzene	103-65-1	0.002	
Styrene	100-42-5	0.002	
1,1,1,2-Tetrachloroethane	630-20-6	0.002	
1,1,2,2-Tetrachloroethane	79-34-5	0.002	
Tetrachloroethene	127-18-4	0.0003	
Toluene	108-88-3	0.002	
1,2,3-Trichlorobenzene	87-61-6	0.002	
1,2,4-Trichlorobenzene	120-82-1	0.002	
1,1,1-Trichloroethane	71-55-6	0.0003	
1,1,2-Trichloroethane	79-00-5	0.0002	
Trichloroethene	79-01-6	0.0012	
Trichlorofluoromethane	75-69-4	0.002	
1,2,3-Trichloropropane	96-18-4	0.002	
1,2,4-Trimethylbenzene	95-63-6	0.002	
1,3,5-Trimethylbenzene	108-67-8	0.002	
Vinyl chloride	75-01-4	0.0018	
o-Xylene	95-47-6	0.0005	
m-Xylene	108-38-3	0.0005	
p-Xylene	106-42-3	0.0005	

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Page III

AQUEOUS OR LIQUID MATRIX

1. Receive sample.
2. Label sample.
3. Log in sample logbook, record what analysis required.
4. Determine what analysis needs to be run on the sample.
   a. If metal analysis, preserve with 1:1 nitric acid and/or refrigerate until ready to prepare for analysis. Go to metal analysis.
   b. If organic analysis, refrigerate to preserve sample for analysis on GC/MS.
   c. Any other analysis, refrigerate to preserve sample.
5. For every ten samples of metal analysis the lab will run a duplicate, and a spike.
6. For every sample run for organic analysis the surrogate and internal standards will be added to run quality control.

SOIL MATRIX

1. Receive sample.
2. Label sample.
3. Log in sample logbook, record what analysis.
4. Determine what analysis needs to be run on the sample.
   a. If metal analysis, preserve with nitric acid and/or refrigerate until ready to prepare for analysis. Go to metal analysis.
   b. If organic analysis, refrigerate until ready to prepare for analysis on GC/MS.
   c. Any other analysis refrigerate to preserve sample.
5. For every ten samples of metal analysis the lab will run a duplicate, and a spike.

METAL ANALYSIS

Digestion Procedure:

a. Weigh out 1 to 2 gram of sample (soil) in a conical beaker. Record wt.

b. Add 10 ml of 1:1 HNO_3, mix the slurry, cover with watch glass. Heat the sample to 90°C and reflux for 10 to 15 minutes without boiling. Allow sample to cool, add 5 ml of concentrated HNO_3 replace the watch glass and reflux for 30 minutes. Repeat this last step to ensure complete oxi-

TABLE 2

REQUIRED CONTAINERS, PRESERVATION AND HOLDING TIMES

NAME	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME
ORGANIC TESTS			
Extractables (Including phthalates, nitroaromatics, PCB's, nitrosamines organochlorine pesticides, isopherone, polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons and TCDD)	G, teflon-lined caps	Cool, 4°C 0.008% $Na_2S_2O_3$	7 days (until extraction) 30 days (after extraction)
Extractables (phenols)	G, teflon-lined caps	Cool, 4°C H_2SO_4 to pH < 2 0.008% $Na_2S_2O_3$	7 days (until extraction) 30 days (after extraction)
Purgeables (halocarbons and aromatics)	G, teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$	14 days
Purgeables (Acrolein and Acrylonitrile)	G, teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$	3 days
Orthophosphate	P,G	Filter on site Cool, 4°C	48 hours
Pesticides	G, teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$	7 days (until extraction) 30 days (after extraction)

TABLE 2

REQUIRED CONTAINERS, PRESERVATION AND HOLDING TIMES

NAME	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME
ORGANIC TESTS			
Phenols	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Phosphorous, elemental	G	Cool, 4°C	48 hours
Phosphorous, total	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days

ADDENDUM II

ADDENDUM II

3.0 INTERFERENCES

3.1 Interferences purged or coextracted from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The Gas Chromatograph will be checked daily to ensure freedom from interferences, under the analysis condition, by analyzing method blanks.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A field blank prepared from reagent Type II water and carried through the sampling and handling protocol, will serve as a check for this contamination.

3.3 Cross-contamination can occur whenever high-level, and low-level samples are analyzed sequentially. When high concentrated samples are analyzed, a reagent water analysis will follow to check contamination. The purge and trap system will be baked out 10 minutes longer and cleaned out after high level samples.

3.4 The laboratory has two separate areas for volatiles. The Gas Chromatograph is located away from the solvent extraction area.

3.5 Impurities in the purge gas, and from organic compounds outgasing from the plumbing ahead of the trap, account for the majority of contamination problems. The laboratory purge gas is certified against impurities. The laboratory does not out-gas before the trap or after. The laboratory demonstrates the freedom from contamination by running calibration and reagent blank analysis. All components on the purging device are TFE plastic.

5.1 The reagent Type II water listed on Table 1 will be used in all analysis, and will be monitored daily.

5.3 Secondary Dilution Standards: The secondary dilution standards will be prepared daily at concentrations of .4ul/ml to 20ul/ml, and will be stored with no headspace for volatiles. All compounds listed in Table 1 will be in the secondary dilution.

5.4 Calibration standards: All compounds listed in Table 1 will be included in all six concentration levels (.4ul/ml, 1ul/ml, 3ul/ml, 5ul/ml, 10ul/ml, 20ul/ml).

5.4.7 Aqueous standards are not stable and will be discarded after 1 hour, or will be sealed and stored for only 24 hours.

5.5 Internal standards will be used.

6.0 See Page III (Cond.)

7.2 Initial calibration for purge-and-trap procedure.

7.2.1 Recommended GC/MS operating conditions

Electron energy:	70 volts (nominal).
Mass range:	35-260 amu.
Scan time:	To give 5 scans/peak but not to exceed 7 sec/scan.
Initial column temperature:	45°C.
Initial column holding time:	3 minutes
Column temperature program:	8°C/minutes
Final column temperature:	220°C.
Final column holding time:	15 minutes
Injector temperature:	200-225°C.
Source tmeperature:	According to manufacturer's specifications.
Transfer line temperature:	250-300°C.
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec.

7.2.2 Each GC/MS system must be hardware-tuned to meet the criteria in Table 3 for a 50-ng injection or purging of 4-bromofluorobenzene (2-uL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.2.3 Assemble a purge-and-trap device that meets the specifications. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily for 10 min while backflushing at 180°C with the column at 220°C.

7.2.4 The purge-and-trap device is connected to a gas chromatograph.

7.2.5 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device (use freshly prepared stock solutions when preparing the calibration standards for the initial calibration.)

Add 5.0 mL of water to the purging device. The water is added to the purging device using a 5-mL glass syringe fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of the 20-gauge needle. Next, using a 10-uL or 25-uL micro-syringe equipped with a long needle, take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards. Add the aliquot of calibration solution directly to the reagent water in the purging device by inserting the needle through the sample inlet. When discharging the contents of the micro-syringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water. Similarly, add 10 uL of the internal standard solution. Close the 2-way syringe valve at the sample inlet.

7.2.7 Tabulate the area response of the characteristic ions (see Table 1) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

7.2.8 The average RF must be calculated for each compound. A system performance check will be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor of 80%. These compounds are chloromethane, 1,1-dichlorcethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). Examples of these occurrences are:

7.2.8.1 Chloromethane - This compound is the most likely compound to be lost if the purge flow is to fast.

7.2.8.2 Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

7.2.8.3 Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.9 Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs).

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

where:

RSD = relative standard deviation.

$\bar{x}$ = mean of 5 initial RFs for a compound.

SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N - 1}}$$

The %RSD for each individual CCC should be less than 30 percent. This criterion must be met in order for the individual calibration to be valid. The CCCs are:

1,1-Dichloroethene,
Chloroform,
1,2-Dichloropropane,
Toluene,
Ethylbenzene, and
Vinyl chloride.

7.3 Daily GC/MS calibration

7.3.1 Prior to the analysis of samples, inject or purge 50-ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hour shift.

7.3.2 The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC and CCC.

7.3.3 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum response factor for volatile SPCCs is 0.300 (0.250 for Bromoform). Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.3.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Step 7.2.9 are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\overline{RF}_I - RF_c}{\overline{RF}_I} \times 100$$

where:

$\overline{RF}_I$ = average response factor from initial calibration.

RF_c = response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid. If the criterion is not met (> 25% difference), for any one CCC, corrective action MUST be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins.

7.3.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two (- 50% to + 100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

7.4.3.1 Low-level method - This is designed for samples containing individual purgeable compounds of < 1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples.

7.4.3.1.1 Use a 5-g sample if the expected concentration is < 0.1 mg/kg or a 1-g sample for expected concentrations between 0.1 and 1 mg/kg.

7.4.3.1.2 The GC/MS system is set up as in Steps 7.4.1.2-7.4.1.4. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature.

7.4.3.1.3 Remove the plunger from a 5-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of surrogate spiking solution and internal standard solution to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together.) The addition of 10 uL of the surrogate spiking solution to 5 g of sediment/soil is equivalent to 50 ug/kg of each surrogate standard.

7.4.3.1.4 The sample (for volatile organics) consists of the <u>entire</u> contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Step 7.4.3.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.4.3.1.5 Determine the percent moisture of the soil/sediment sample. This includes waste samples that are amenable to moisture determination. Other wastes should be reported on a wet-weight basis. Immediately after weighing the sample, weigh (to 0.1 g) 5-10 g of additional sediment/soil into a tared crucible.

Dry the contents of the crucibles overnight at 105°C. Allow to cool in a desiccator and reweigh the dried contents. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\% \text{ moisture} = \frac{\text{grams of sample - grams of dry sample}}{\text{grams of sample}} \times 100$$

7.4.3.1.6 Add the spiked water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

NOTE: Prior to the attachment of the purge device, the procedures in Steps 7.4.3.1.4 and 7.4.3.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

7.4.3.1.7 Heat the sample to 40°C ± 1°C and purge the sample for 11.0 ± 0.1 minute.

7.4.3.1.8 Proceed with the analysis. Use 5 mL of the same water as in the reagent blank. If saturated peaks occurred or would occur if a 1-g sample were analyzed, the medium-level method must be followed.

7.4.3.1.9 For low-level sediment/soils add 10 uL of the matrix spike solution to the 5 mL of water (Step 7.4.3.1.3). The concentration for a 5-g sample would be equivalent to 50 ug/kg of each matrix spike standard.

7.4.3.2 High-level method - The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e. petroleum and coke wastes) that are insoluble in methanol are diluted with reagent tetraglyme or possibly polyethylene glycol (PEG). An aliquot of the extract is added to reagent water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of >1.0 mg/kg should be analyzed by this method.

7.4.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol weigh 4 g (wet weight) of sample into a tared 20-mL vial. Use a top-

loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Step 7.4.3.1.5. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10-mL volumetric flask.

8.0 QUALITY CONTROL

8.1 Each laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements for this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control reference sample must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst will demonstrate, through the analysis of a calibration blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank will be processed as a safeguard against chronic laboratory contamination. The blank samples will be carried through all stages of sample preparation and measurement.

8.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard will be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still useable, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

8.4 Required instrument QC is found in the following section:

8.4.1 The GC/MS system must be tuned to meet the BFB specifications in Step 7.2.2.

8.4.2 There must be an initial calibration of the GC/MS system as specified in Step 7.2.

8.4.3 The GC/MS system must meet the SPCC criteria specified in Step 7.3.3 and the CCC criteria in Step 7.3.4, each 12 hours.

8.5 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.5.1 A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 10 ug/mL in methanol. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions.

8.5.2 Prepare a QC reference sample to contain 20 ug/L of each analyte by adding 200 uL of QC reference sample concentrate to 100 mL of water.

8.5.3 Four 5-mL aliquots of the well-mixed QC reference sample are analyzed according to the method.

8.5.4 Calculate the average recovery ($\bar{x}$) in ug/L, and the standard deviation of the recovery (s) in ug/L, for each analyte using the four results.

8.5.5 For each analyte compare s and $\bar{x}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and $\bar{x}$ for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{x}$ falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined.

8.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Step 8.5.6.1 or 8.5.6.2.

8.5.6.1 Locate and correct the source of the problem and repeat the test for all analytes beginning with Step 8.5.2.

8.5.6.2 Beginning with Section 8.5.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Step 8.5.2.

8.6 The laboratory must, on an ongoing basis, analyze a reagent blank and a spiked replicate for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are

present, replicate samples may be appropriate in place of spiked replicates. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.6.1 The concentration of the spike in the sample will be determined as follows:

8.6.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike will be at that limit or 1 to 5 times higher than the background concentration determined in Step 8.6.2, whichever concentration would be larger.

8.6.1.2 If the concentration of a specific analyte in a water sample is not being checked against a specific limit, the spike should be at 20 ug/L or 1 to 5 times higher than the background concentration determined in Step 8.6.2, whichever concentration would be larger. For other matrices, recommended spiking concentration is 10 times the PQL.

8.6.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each analyte. If necessary, prepare a new QC reference sample concentrate (Step 8.5.1) appropriate for the background concentration in the sample. Spike a second 5-mL sample aliquot with 10 uL of the QC reference sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as $100(A-B)\%/T$, where T is the known true value of the spike.

8.6.3 Compare the percent recovery(p) for each analyte in a water sample with the corresponding QC acceptance criteria found in Table 6. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1. If spiking was performed at a concentration lower than 20 ug/L, the analyst must use either the QC acceptance criteria presented in Table 6, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of an analyte: (1) Calculate accuracy (x') using the equation found in Table 7, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 7, substituting x' for $\bar{x}$; (3) calculate the range for recovery at the spike concentration as $(100x'/T) \pm 2.44(100S'/T)\%$.

8.6.4 If any individual p falls outside the designated range for recovery, that analyte has failed the acceptance criteria. A check standard containing each analyte that failed the criteria must be analyzed as described in Step 8.7.

8.7 If any analyte in a water sample fails the acceptance criteria for recovery in Step 8.6, a QC reference sample containing each analyte that failed must be prepared and analyzed.

8.7.1 Prepare the QC reference sample by adding 10 uL of the QC reference sample concentrate (Step 8.5.1 or 8.6.2) to 5 mL of reagent water. The QC reference sample needs only to contain the analytes that failed criteria in the test in Step 8.6.

8.7.2 Analyze the QC reference sample to determine the concentration measured (A) of each analyte. Calculate each percent recovery (p_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.7.3 Compare the percent recovery (p_s) for each analyte with the corresponding QC acceptance criteria found in Table 6. Only analytes that failed the test in Step 8.6 need to be compared with these criteria. If the recovery of any such analyte falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem must be immediately identified and corrected. The result for that analyte in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.8 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of five spiked samples (of the same matrix) as in Section 8.6, calculate the average percent recovery ($\bar{p}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{p} - 2s_p$ to $\bar{p} + 2s_p$. If $\bar{p} = 90\%$ and $s_p = 10\%$, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis (e.g. after each five to ten new accuracy measurements).

8.9 To determine acceptable accuracy and precision limits for surrogate standards the following procedure will be performed.

8.9.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

8.9.2 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (p) and standard deviation of the percent recovery (s) for each of the surrogates.

8.9.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard.

$$\text{Upper Control Limit (UCL)} = p + 3s$$
$$\text{Lower Control Limit (LCL)} = p - 3s$$

8.9.4 For aqueous and soil matrices, these laboratory established surrogate control limits should, if applicable, be compared with the control limits listed in Table 8. The limits given in Table 8 are multi-laboratory performance based limits for soil and aqueous samples, and therefore, the single-laboratory limits established in Step 8.9.3 must fall within those given in Table 8 for these matrices.

8.9.5 If recovery is not within limits, the following procedures are required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

8.9.6 At a minimum, each laboratory will update surrogate recovery limits on a matrix-by-matrix basis, annually.

FORM 1

ANALYTICAL LABORATORY SERVICES

A DIVISION OF M C CHEMICAL
P.O. Box 1926 · Rockford, IL 61110
815/964-7570

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

COMPONENT	CONC. SPK ADD	SAMPLE RESULTS	CONC. MS	% REC	CONC. MSD	% REC

Form 2

ANALYTICAL LABORATORY SERVICES

A DIVISION OF M C CHEMICAL
P.O. Box 1926 · Rockford, IL 61110
815/964-7570

SOIL SURROGATE PERCENT RECOVERY SUMMARY

Sample Number	TOLUENE-D_8 81-117%	BROMOFLUORO-BENZENE 74-121%	1,2 DICHLORO-ETHANE-D_4 76-121%

US4569

ANALYTICAL LABORATORY SERVICES
A DIVISION OF M C CHEMICAL
P.O. Box 1926 · Rockford, IL 61110
815/964-7570

VOLATILE COMPOUNDS

TCLP MS / SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

CASE NO._____ SAMPLE NO._____

COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS RPD	QC LIMITS RECOVERY
Benzene									
Carbon Tetrachloride									
Chlorobenzene									
Chloroform									
1,4 Dichlorobenzene									
1,2 Dichloroethane									
1,1 Dichloroethylene									
Tetrachloroethylene									
Trichloroethylene									
Vinyl Chloride									
2-Butanone									

Form 3

ANALYTICAL LABORATORY SERVICES
A DIVISION OF M C CHEMICAL
P.O. Box 1926 · Rockford, IL 61110
815/964-7570

SEMI VOLATILE

TCLP MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

CASE NO._____ SAMPLE NO._____

COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS RPD	QC LIMITS RECOVERY
O-Cresol									
M-Cresol									
P-Cresol									
1,4 Dichlorobenzene									
2,4 Dinitrotoluene									
Hexachlorobenzene									
Hexachlorobutadiene									
Hexachloroethane									
Nitrobenzene									
Pentachlorophenol									
Pyridine									
2,4,5 Trichlorophenol									
2,4,6 Trichlorophenol									

ANALYTICAL LABORATORY SERVICES
A DIVISION OF M C CHEMICAL
P.O. Box 1926 · Rockford, IL 61110
815/964-7570

PESTICIDES & HERBICIDES

TCLP MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

CASE NO._____ SAMPLE NO._____

COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS RPD	QC LIMITS RECOVERY
Chlordane									
Heptachlor									
Heptachlor Epoxide									
Lindane									
Methoxychlor									
Toxaphene									
2,4-D									
2,4,5-T P									

ANALYTICAL LABORATORY SERVICES

A DIVISION OF M C CHEMICAL
P.O. Box 1926 · Rockford, IL 61110
815/964-7570

	CAS No.	ADL	RESULTS
Methylene chloride	75-09-2	0.005	
Naphthalene	91-20-3	0.002	
n-Propylbenzene	103-65-1	0.002	
Styrene	100-42-5	0.002	
1,1,1,2-Tetrachloroethane	630-20-6	0.002	
1,1,2,2-Tetrachloroethane	79-34-5	0.002	
Tetrachloroethene	127-18-4	0.0003	
Toluene	108-88-3	0.002	
1,2,3-Trichlorobenzene	87-61-6	0.002	
1,2,4-Trichlorobenzene	120-82-1	0.002	
1,1,1-Trichloroethane	71-55-6	0.0003	
1,1,2-Trichloroethane	79-00-5	0.0002	
Trichloroethene	79-01-6	0.0012	
Trichlorofluoromethane	75-69-4	0.002	
1,2,3-Trichloropropane	96-18-4	0.002	
1,2,4-Trimethylbenzene	95-63-6	0.002	
1,3,5-Trimethylbenzene	108-67-8	0.002	
Vinyl chloride	75-01-4	0.0018	
o-Xylene	95-47-6	0.0005	
m-Xylene	108-38-3	0.0005	
p-Xylene	106-42-3	0.0005	

TABLE 3.
BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

TABLE 3.
BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

TABLE 6.
CALIBRATION AND QC ACCEPTANCE CRITERIA[a]

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for x (ug/L)	Range p,p_s (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.4	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.4	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethyl benzene	11.8-28.2	7.5	17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.5-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Q = Concentration measured in QC check sample, in ug/L.
s = Standard deviation of four recovery measurements, in ug/L.
x = Average recovery for four recovery measurements, in ug/L.
p, p_s = Percent recovery measured.
D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

TABLE 6.
CALIBRATION AND QC ACCEPTANCE CRITERIA[a]

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for x (ug/L)	Range p, p_s (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.4	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.4	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethyl benzene	11.8-28.2	7.5	17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.5-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Q = Concentration measured in QC check sample, in ug/L.
s = Standard deviation of four recovery measurements, in ug/L.
x = Average recovery for four recovery measurements, in ug/L.
p, p_s = Percent recovery measured.
D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

METHOD 5030

METHOD 5030

1.0 SCOPE AND APPLICATION

1.1 This method describes sample preparation and extraaction for the analysis of volatile organics by a purge-and-trap procedure. The gas chromatographic determinative steps are found in Methods 8010, 8015, 8020, and 8030. Although applicable to Method 8240, the purge-and trap procedure is already incorporated into Method 8240.

1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200°C (vapor pressure is approximately equal to mm Hg @ 25°C) and are insoluble or silightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system.

1.4 This method also describes the preparation of water-miscible liquids, solids, wastes, and soil/sediments for analysis by the purge-and-trap procedure.

2.0 SUMMARY OF METHOD

2.1 The purge-and-trap process: An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a specially designed purging chamber. It is then analyzed by purge-and-trap GC following the water method.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A field reagent blank prepared from reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-level and low-level samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination; therefore, frequent bake-out and purging of the entire system may be required.

3.4 The laboratory where volatile analysis is preformed should be completely free of solvents.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes: 10-uL, 25-uL, 100-uL, 250-uL, 500-uL, and 1,000 uL: These syringes should be equipped with a 20-gauge (0.006-in I.D.) needle having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device. The needle length will depend upon the dimensions of the purging device employed.

4.2 Syringe valve: Two-way, with Luer ends (three each), if applicable to the purging device.

4.3 Syringe: 5-mL, gas-tight with shutoff valve.

4.4 Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g.

4.5 Glass scintillation vials: 20-mL, with screw-caps and Teflon liners or glass culture tubes with a screw-cap and Teflon liner.

4.6 Voulumetric flasks: 10-mL and 100-mL, class A with ground-glass stoppers.

4.7 Vials: 2-mL, for GC autosampler.

4.8 Spatula: Stainless steel.

4.9 Disposable pipets: Pasteur.

4.10 Purge-and-trap device: The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

4.10.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3-mm at the origin. The purge gas must be introduced no more than 5mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be used, provided equivalent performance is demonstrated.

4.10.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (See Figures 2 and 3). If it is not necessary to analyze for dichlorodifluromethane or other fluorocarbons of similar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°c by backflushing with an inert gas flow of at least 20mL/min. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

4.10.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. The desorber design illustrated in Figures 2 and 3 meet these criteria.

4.10.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as shown in Figures 4 and 5.

4.10.5 Trap Packing Materials

4.10.5.1 2,6-Diphenylene oxide polymer: 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.10.5.2 Methyl silicone packing: OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

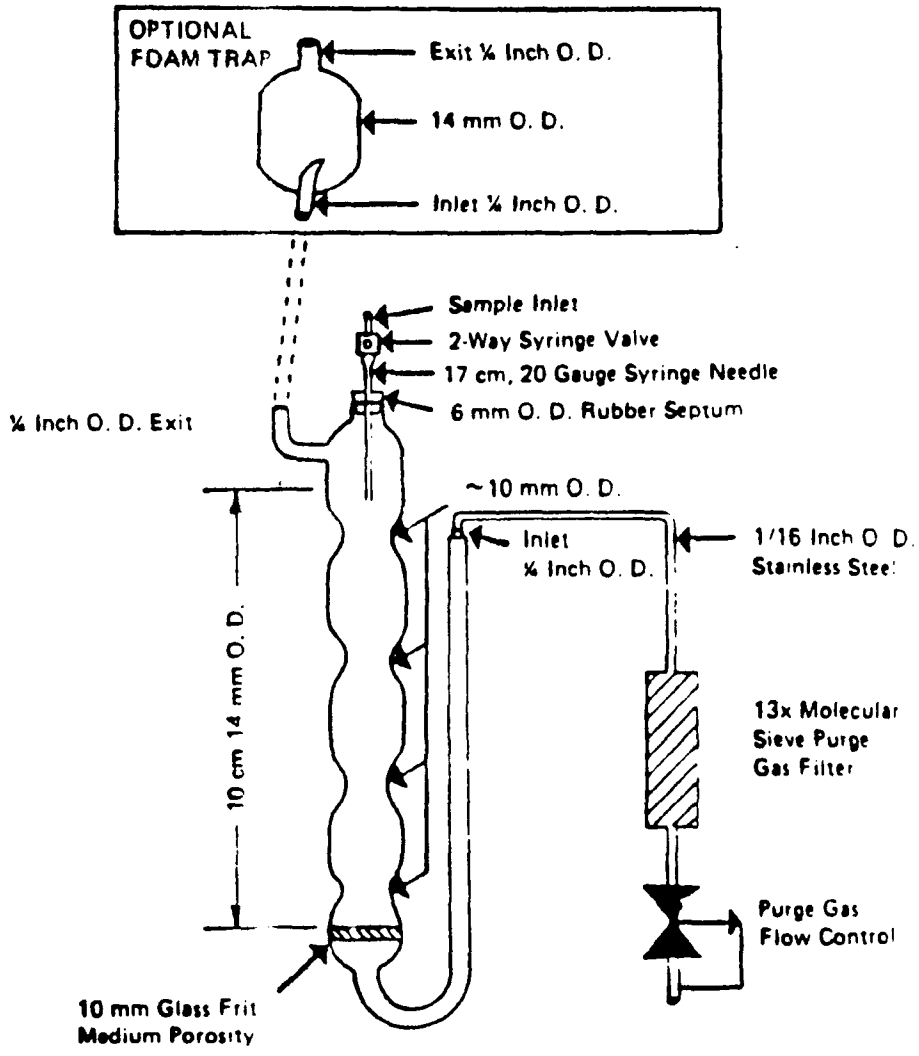


Figure 1. Purging chamber.

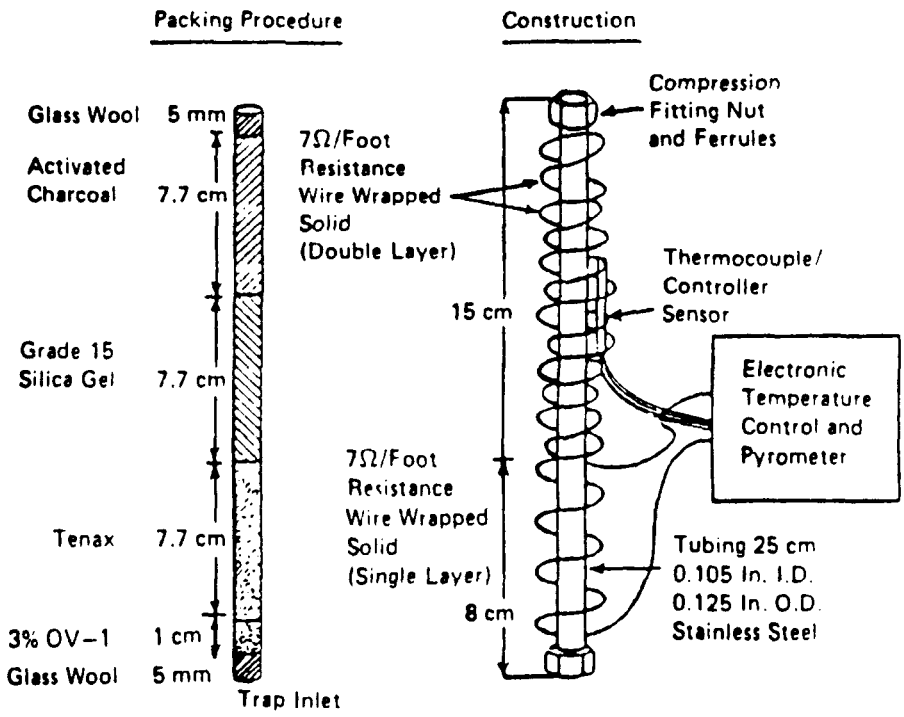


Figure 2. Trap packings and construction for Method 8010.

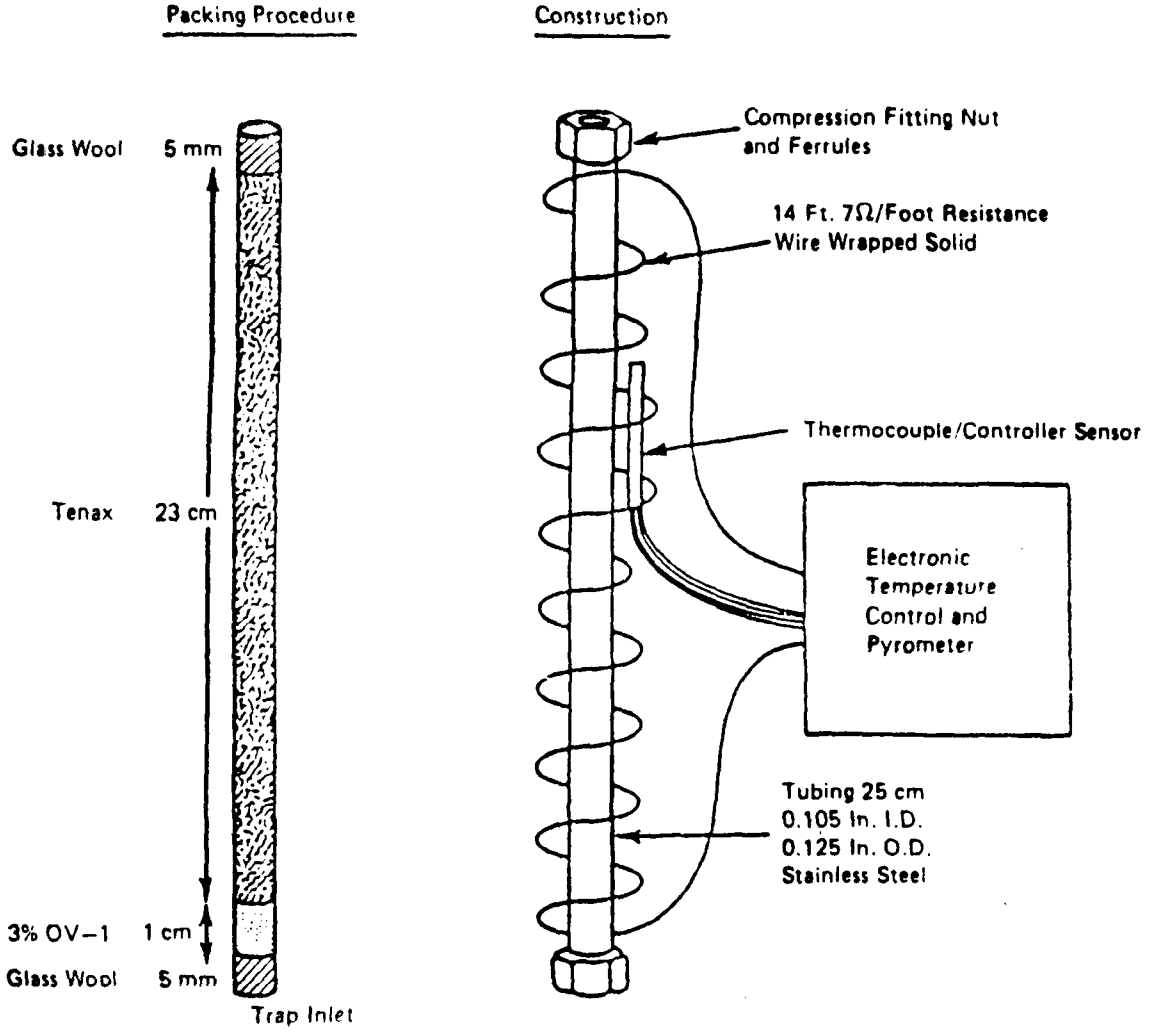


Figure 3. Trap packing and construction for Methods 8020 and 8030.

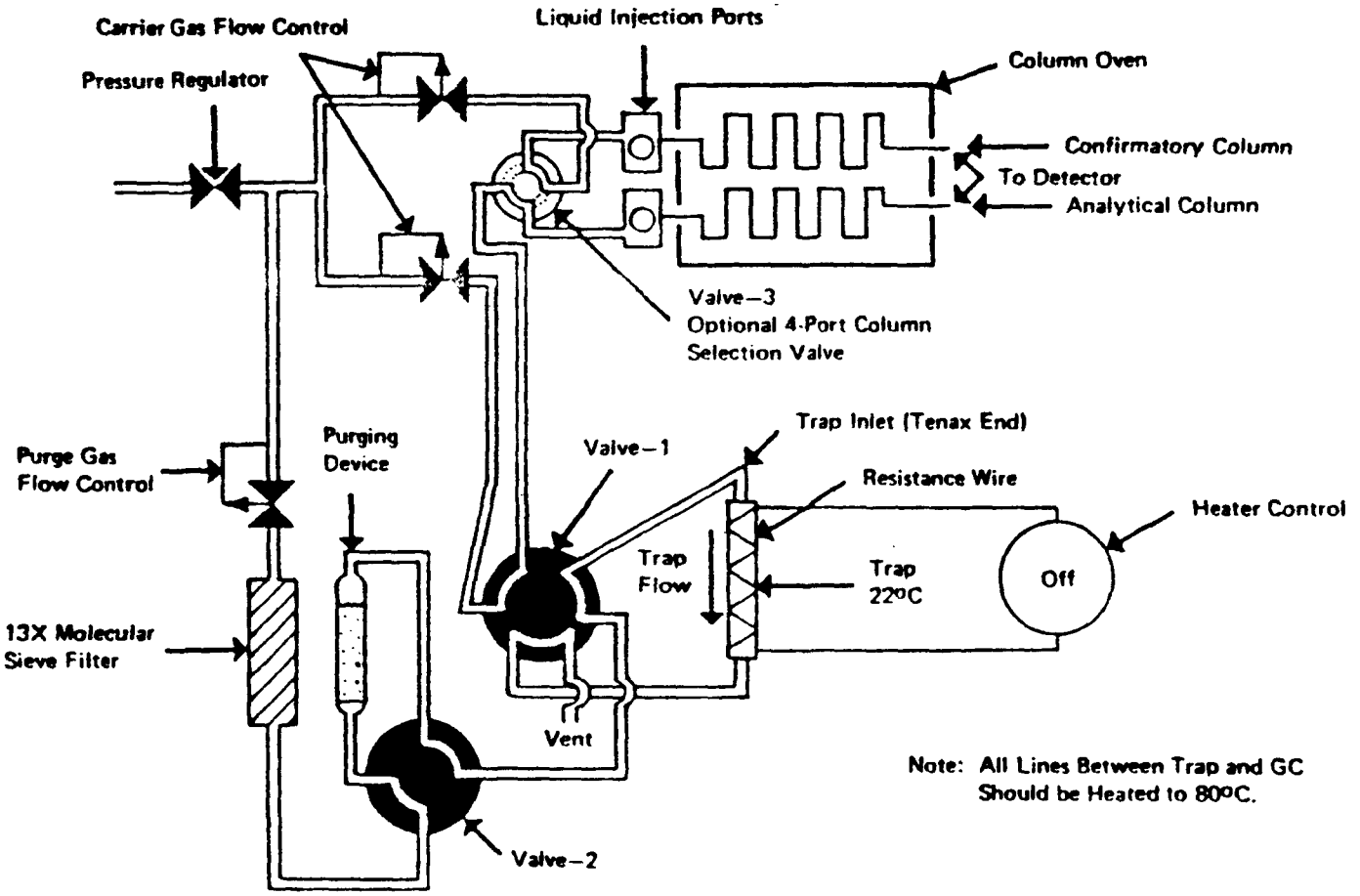


Figure 4. Purge-and-trap system, purge-sorb mode, for Methods 8010, 8020, and 8030.

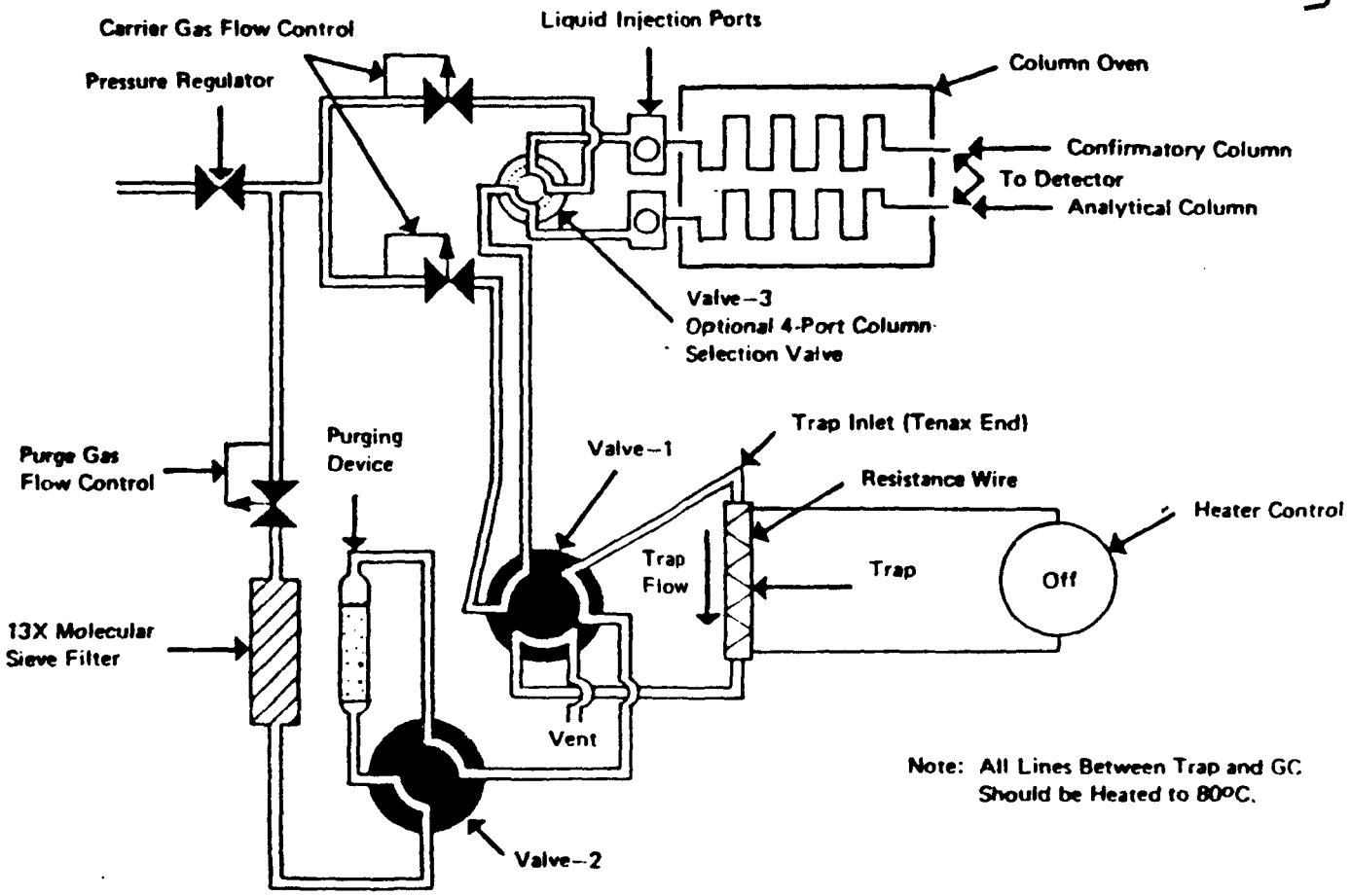


Figure 5. Purge-and-trap system, desorb mode, for Methods 8010, 8020, and 8030.

4.10.5.3 Silica gel: 35/60 mesh, Davison, grade 15 or equivalent.

4.10.5.4 Coconut charcoal: Prepare from Barnebey Cheney, CA-580-26 lot #M-2649, by crushing through 26 mesh screen.

4.11 Heater or heated oil bath: Should be capable of maintaining the purging chamber to within 1°C over a temperature range from ambient to 100°C.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit of the compounds of interest.

5.1.1 Reagent water may be generated by passing trap water through a carbon filter bed containing about 500 g of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

5.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the water temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hr. While still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

5.2 Methanol: Pesticide quality or equivalent. Store away from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Initial calibration: Prior to using this introduction technique for any GC method, the system must be calibrated. General calibration procedures are discussed in Method 8000, Section, 7.4, while the specific determinative methods and Method 3500 give details on preparation of standards.

7.1.1 Assemble a purge-and-trap device that meets the specification in Section 4.10. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily for 10 min while backflushing at 180°C with the column at 220°C.

7.1.2 Connect the purge-and-trap device to a gas chromatograph.

7.1.3 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. Add 5.0 mL of reagent water to the purging device. The reagent water is added to the purging device using a 5-mL glass syringe fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of the 20-gauge needle. Next, using a 10-uL or 25-uL microsyringe equipped with a long needle (Paragraph 4.1), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards. Add the aliquot of calibration solution directly to the reagent water in the purging device by inserting the needle through the sample inlet. When discharging the contents of the micro-syringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water. Similarly, add 10 uL of the internal standard solution. Close the 2-way syringe valve at the sample inlet.

7.1.4 Carry out the purge-and-trap analysis procedure using the specific conditions given in Table 1.

7.1.5 Calculate response factors or calibration factors for each analyte of interest using the procedure described in Method 8000, Section 7.4.

7.1.6 The average RF must be calculated for each compound. A system performance check should be made before this calibration curve is used. If the purge-and-trap procedure is used with Method 8010, the following five compounds are checked for a mimimun average response factor: chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane; and chlorobenzene. The mimimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds typically have RFs of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.1.6.1 Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.

7.1.6.2 Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purgeflow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

7.1.6.3 Tetrachloroethane and 1,1-dichloroethane: These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

TABLE 1. PURGE-AND-TRAP OPERATING PARAMETERS

	Analysis Method			
	8010	8015	8020	8030
Purge gas	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium	Nitrogen or Helium
Purge gas flow rate (mL/min)	40	20	40	20
Purge time (min)	11.0 ± 0.1	15.0 ± 0.1	12.0 ± 0.1	15.0 ± 0.1
Purge temperature (°C)	Ambient	85 ± 2	Ambient	85 ± 2
Desorb temperature (°C)	180	180	180	180
Backflush inert gas flow (mL/min)	20-60	20-60	20-60	20-60
Desorb time (min)	4	1.5	4	1.5

7.2 On-going calibration: Refer to Method 8000, Sections 7.4.2.3 and 7.4.3.4 for details on continuing calibration.

7.3 Sample preparation:

7.3.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Two screening techniques that can be utilized are: The use of an automated headspace sampler (modified Method 3810), interfaced to a gas chromatograph (GC), equipped with a photo ionization detector (PID), in series with an electrolytic conductivity detector (ECD); and extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC with a FID and/or an ECD.

7.3.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.3.1.3 Assemble the purge-and-trap device. The operating conditions for the GC are given in Section 7.0 of the specific determinative method to be employed.

7.3.1.4 Daily GC calibration criteria must be met (Method 8000, Section 7.4) before analyzing samples.

7.3.1.5 Adjust the purge gas flow rate (nitrogen or helium) to that shown in Table 1, on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

7.3.1.6 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample intergrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20-mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hrs. Care must be taken to prevent air from leaking into the syringe.

7.3.1.7 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.3.1.7.1 Dilutions may be made in volumetric flasks (10-mL to 100-mL). Select the volumetric flask that will allow for the necessary for extremely large dilutions.

7.3.1.7.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

7.3.1.7.3 Inject the proper aliquot of samples from the syringe prepared in Paragraph 7.3.1.5 into the flask. Aliquots of less than 1-mL are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions

7.3.1.7.4 Fill a 5-mL syringe with the diluted sample as in Paragraph 7.3.1.5.

7.3.1.8 Add 10.0 uL of surrogate spiking solution found in each determinative method, Section 5.0) and, if applicable, 10 uL of internal standard spiking solution through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. Matrix spiking solutions, if indicatedm should be added (10-uL) to the sample at this time.

7.3.1.9 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

7.3.1.10 Close both valves and purge the sample for the time and at the temperature specified in Table 1.

7.3.1.11 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program and GC data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with inert gas between 20 and 60 mL/min for the time specified in Table 1.

7.3.1.12 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5-mL flushes of reagent water (or methanol followed by reagent water) to avoid carryover of pollutant compounds into subsequent analyses.

7.3.1.13 After desorbing the sample, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 sec; then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained

at 180°C for Methods 8010 and 8020, and 210°C for Methods 8015 and 8030. Trap temperatures up to 220°C may be employed; however, the higher temperature will shorten the useful life of the trap. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.3.1.14 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated response from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.3.1.15 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Method 8000 and the specific determinative method for details on caculation analyte response.

7.3.2 Water-miscible liquids

7.3.2.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with reagent water.

7.3.2.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100-mL volumetric flask and diluting to volume with reagent water. Transfer immediately to a 5-mL gas-tight syringe.

7.3.2.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with reagent water by adding at least 20 uL, but not more than 100-uL of liquid sample. The sample is ready for addition of surrogate and, if applicable, internal and matrix spiking standards.

7.3.3 Sediment/soil and waste samples: It is highly recommended that all samples of this type be screened prior to the purge-and-trap GC analysis. These samples may contain percent quantities of purgeable organics that will contaminte the purge-and-trap system, and require extensive cleanup and instrument downtime. See Paragraph 7.3.1.1 for recommended screening techniques. Use the screening data to determine whether to use the low-level method (0.005-1 mg/kg) or the high-level method (1mg/kg).

7.3.3.1 Low-level method: This is designed for samples containing individual purgeable compounds of < 1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples.

7.3.3.1.1 Use a 5-g sample if the expected concentration is 0.1 mg/kg or a 1-g sample for expected concentrations between 0.1 and 1 mg/kg.

7.3.3.1.2 The GC system should be set up as in Section 7.0 of the specific determinative method. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature for Methods 8010 and 8020.

7.3.3.1.3 Remove the plunger from a 5-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of surrogate spiking solution and internal standard solution to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together). Matrix spiking solutions, if indicated, should be added (10 ul) to the sample at this time.

7.3.3.1.4 The sample (for volatile organics) consists of the entire contents for the sampler container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Paragraph 7.3.3.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.3.3.1.5 In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination. Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

7.3.3.1.6 Add the spiked reagent water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

NOTE: Prior to the attachment of the purge device, steps 7.3.3.1.4 and 7.3.3.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

7.3.3.1.7 Heat the sample to 40°C ± 1°C (Methods 8010 and 8020) or to 85°C ± 2°C (Methods 8015 and 8030) and purge the sample for the time shown in Table 1.

7.3.3.1.8 Proceed with the analysis as outlined in Paragraphs 7.3.1.11-7.3.1.15. Use 5 mL of the same reagent water as in the reagent blank. If saturated pecks occurred or would occur if a 1-g sample were analyzed, the high-level method must be followed.

7.3.3.2 High-level method: The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to reagent water containing surrogate and, if applicable, internal and matrix spiking standards. This is purged at the temperatures indicated in Table 1. All samples with an expected concentration of 1.0 mg/kg should by analyzed by this method.

7.3.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 4 g (wet weight) of sample into a tared 20-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Paragraph 7.3.3.1.5. For waste that is soluble in methanol, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10-mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 mL of methanol into the vial and mark the bottom of the meniscus. Discard this solvent).

7.3.3.2.2 Quickly add 9.0 mL of methanol; then add 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 min.

NOTE: Steps 7.3.3.2.1 and 7.3.3.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.3.3.2.3. Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 mL of reagent methanol to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the dark, prior to analysis.

7.3.3.2.4 The GC system should be set up as in Section 7.0 of the specific determinative method. This should be done prior to the addition of the methanol extract to reagent water.

7.3.3.2.5 Table 2 can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-level analysis to determine the appropriate volume. If the sample was submitted as a high-level sample, start with 100 uL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.3.3.2.6 Remove the plunger from a 5.0-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 uL of internal standard solution. Also add the volume of methanol extract determined in Paragraph 7.3.3.2.5 and a volume of methanol solvent to total 100 uL (excluding methanol in standards).

7.3.3.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

7.3.3.2.8 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 uL of methanol to simulate the sample conditions.

7.3.3.2.9 For a matrix spide in the high-level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution and 1.0 mL of matrix spike solution. Add a 100-uL aliquot of this extract to 5 mL of water for purging (as per Paragraph 7.3.3.2.6).

7.4 Sample analysis:

7.4.1 The samples prepared by this method may be analyzed by Methods 8010, 8015, 8030, and 8240. Refer to these methods for appropriate analysis conditions.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 3500 for sample preparation procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of a reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard through all stages of the sample preparation and measurement.

8.3 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique. Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis. If the fortified samples do not indicate sufficient sensitivity to detect <1 ug/g of the analytes in the sample, then the sensitivity of the instrument should be increased, or the sample should be subjected to additional cleanup.

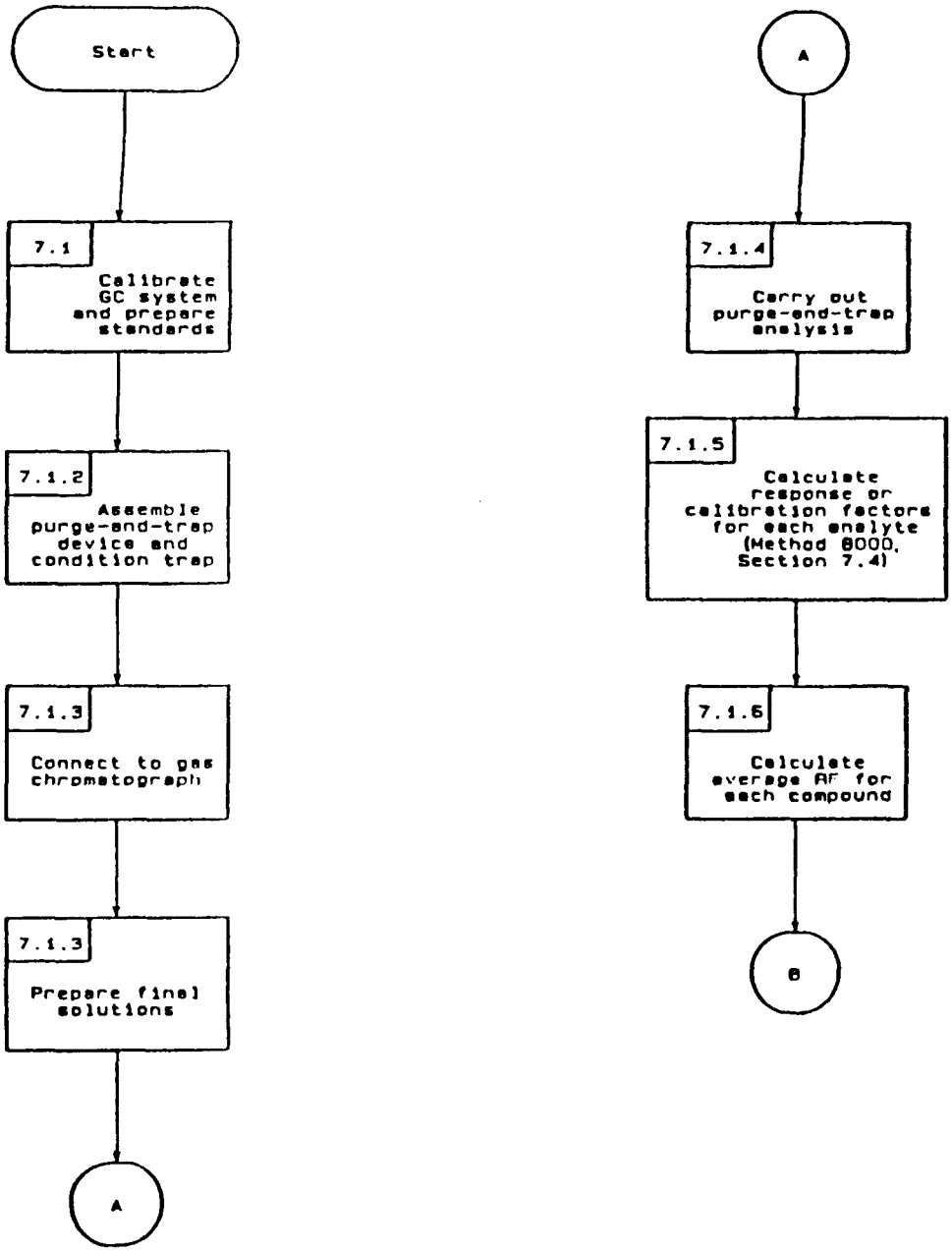
TABLE 2. QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-LEVEL SOILS/SEDIMENTS

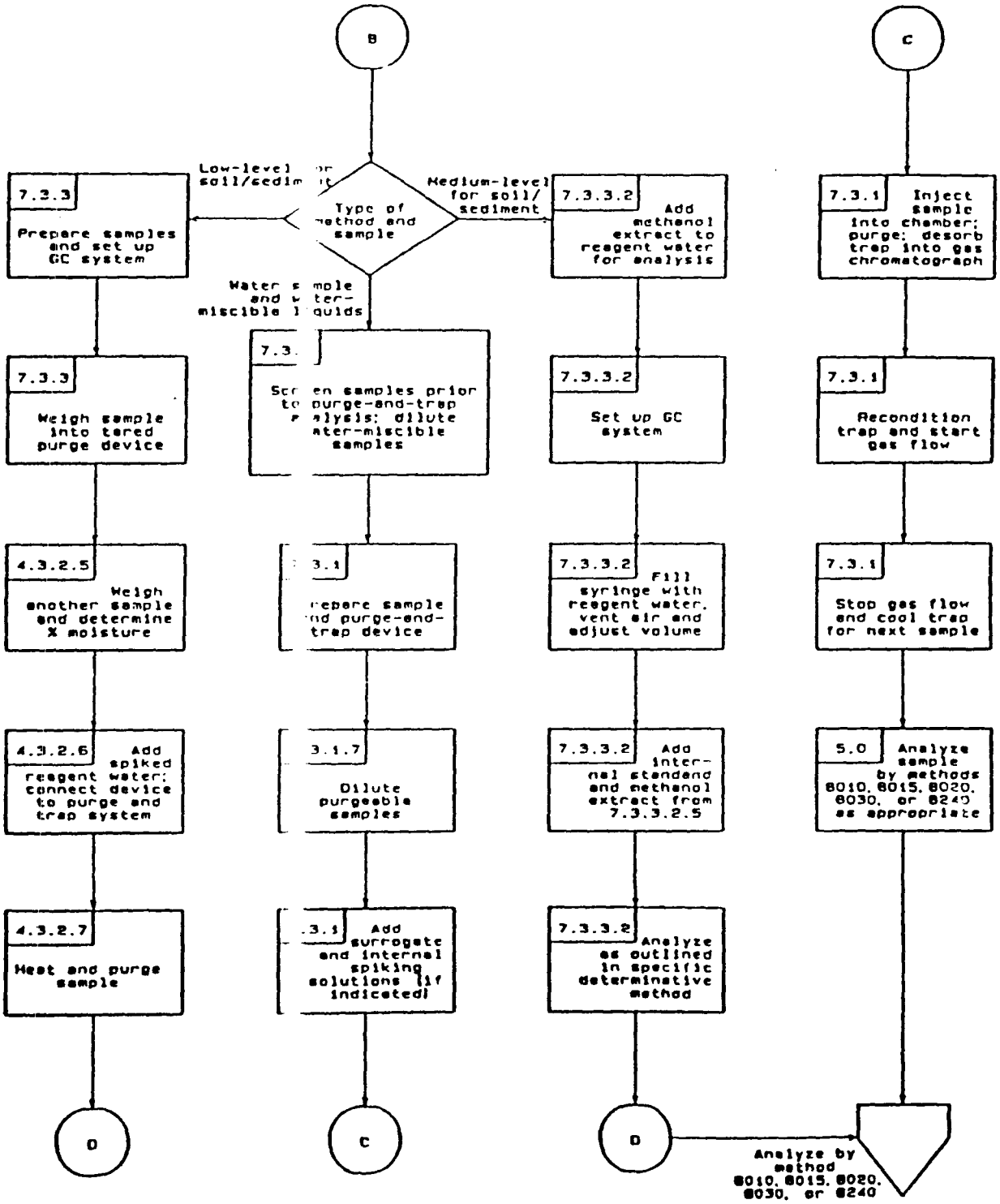
Approximate Concentration Range	Volume of Methanol Extract[a]
500-10,000 ug/kg	100 uL
1,000-20,000 ug/kg	50 uL
5,000-100,000 ug/kg	10 uL
25,000-500,000 ug/kg	100 uL of 1/50 dilution [b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a]The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

[b]Dilute an aliquot of the methanol extract and then take 100 uL for analysis.


Start
7.1
Calibrate GC system and prepare standards
7.1.2
Assemble purge-and-trap device and condition trap
7.1.3
Connect to gas chromatograph
7.1.3
Prepare final solutions
A
A
7.1.4
Carry out purge-and-trap analysis
7.1.5
Calculate response or calibration factors for each analyte (Method 8000, Section 7.4)
7.1.6
Calculate average RF for each compound
B


B
C
Type of method and sample
Low-level soil/sedim
Medium-level for soil/ sediment
Water sample and water-miscible liquids
7.3.3
Prepare samples and set up GC system
7.3.3.2
Add methanol extract to reagent water for analysis
7.3.1
Inject sample into chamber; purge; desorb trap into gas chromatograph
7.3.3
Weigh sample into tared purge device
7.3.
Screen samples prior to purge-and-trap analysis; dilute water-miscible samples
7.3.3.2
Set up GC system
7.3.1
Recondition trap and start gas flow
4.3.2.5
Weigh another sample and determine % moisture
3.1
Prepare sample and purge-and-trap device
7.3.3.2
Fill syringe with reagent water, vent air and adjust volume
7.3.1
Stop gas flow and cool trap for next sample
4.3.2.6
Add spiked reagent water; connect device to purge and trap system
3.1.7
Dilute purgeable samples
7.3.3.2
Add internal standard and methanol extract from 7.3.3.2.5
5.0
Analyze sample by methods 8010, 8015, 8020, 8030, or 8240 as appropriate
4.3.2.7
Heat and purge sample
3.1
Add surrogate and internal spiking solutions (if indicated)
7.3.3.2
Analyze as outlined in specific determinative method
D
C
D
Analyze by method 8010, 8015, 8020, 8030, or 8240

METHOD 8240

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS

METHOD 8240

1.0 SCOPE AND APPLICATION

1.1 Method 8240 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

1.2 Method 8240 can be used to quantify most volatile organic compounds that have boiling points below 200°C [vapor pressure is approximately equal to mm Hg @ 25°C] and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique, however, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Table 1 for a list of compounds, retention times, and their characteristic ions that have been evaluated on a purge-and-trap GC/MS system.

1.3 The practical quantitation limit (PQL) of Method 8240 for an individual compound is approximately 5 ug/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 ug/L for ground water (see Table 2). PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.4 Method 8240 is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.5 To increase purging efficiencies of acrylonitrile and acrolein, refer to Methods 5030 and 8030 for proper purge-and-trap conditions.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection (in limited applications). The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to porvide both qualitative and quantitative information. The chromatographic conditions, as well as typical mass spectrometer operating parameters, are given.

TABLE 1. RETENTION TIMES AND CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Acetone	--	43	58
Acrolein	--	56	55, 58
Acrylonitrile	--	53	52, 51
Benzene	17.0	78	52, 77
Bromochloromethane (I.S.)	9.3	128	49, 130, 51
Bromodichloromethane	14.3	83	85, 129
4-Bromofluorobenzene (surr.)	28.3	95	174, 176
Bromoform	19.8	173	171, 175, 252
Bromomethane	3.1	94	96, 79
2-Butanone	--	72	57, 43
Carbon disulfide	--	76	78
Carbon tetrachloride	13.7	117	119, 121
Chlorobenzene	24.6	112	114, 77
Chlorobenzene-d_5 (I.S.)	--	117	82, 119
Chlorodibromomethane	--	129	208, 206
Chloroethane	4.6		66, 49
2-Chloroethyl vinyl ether	18.6	6	65, 106
Chloroform	11.4	83	85, 47
Chloromethane	2.3	50	52, 49
Dibromomethane	--	93	174, 95
1,4-Dichloro-2-butane	--	75	53, 89
Dichlorodifluoromethane	--	85	87, 50, 101
1,1-Dichloroethane	--	63	65, 83
1,2-Dichloroethane	10.1	62	64, 98
1,2-Dichloroethane-d_4 (surr.)	12.1	65	102
1,1-Dichloroethene	9.0	96	61, 98
trans-1,2-Dichloroethene	10.0	96	61, 98
1,2-Dichloropropane	15.7	63	62, 41
cis-1,3-Dichloropropene	15.9	75	77, 39
trans-1,3-Dichloropropene	17.2	75	77, 39
1,4-Difluorobenzene (I.S.)	19.6	114	63, 88
Ethanol	--	31	45, 27, 46
Ethylbenzene	26.4	106	91
Ethyl methacrylate	--	69	41, 39, 99
2-Hexanone	--	43	58, 57, 100
Iodomethane	--	142	127, 141
Methylene chloride	6.4	84	49, 51, 86
4-Methyl-2-pentanone	--	43	58, 100
Styrene	--	104	78, 103
1,1,2,2-Tetrachloroethane	22.1	83	85, 131, 133
Tetrachloroethene	22.2	164	129, 131, 166
Toluene	23.5	92	91, 65
Toluene-d_8 (surr.)		98	70, 100
1,1,1-Trichloroethane	13.4	97	99, 117
1,1,2-Trichloroethane	17.2	97	83, 85, 99
Trichloroethene	16.5	130	95, 97, 132
Trichlorofluoromethane	8.3	101	103, 66
1,2,3-Trichloropropane	--	75	110, 77, 61
Vinyl acetate	--	43	86
Vinyl chloride	3.8	62	64, 61
Xylene	--	106	91

TABLE 2. PRACTICAL QUANTITATION LIMITS (PQL) FOR VOLATILE ORGANICS[a]

Volatiles	CAS Number	Practical Quantitation Limits[b] Ground water ug/L	Low Soil/Sediment ug/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	100	100
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	100	100
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	50	50
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	50	50
29. 4-Methyl-2-pentanone	108-10-1	50	50
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achieveable. See the following information for further guidance on matrix-dependent PQLs.

[b]PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, PQLs will be higher, based on the % moisture in each sample.

Other Matrices:	Factor[1]
Water miscible liquid waste	50
High-level soil & sludges	125
Non-water miscible waste	500

[1]PQL = [PQL for ground water (Table 2)] x [Factor]. For non-aqueous samples, the factor is on a wet-weight basis.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a specially designed purging chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.

2.3 The purge-and-trap process: An inert gas is bubbled through the solution at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components, which are detected with a mass spectrometer.

3.0 INTERFERENCES

3.1 Interferences purged or coextracted from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of reagent water to check for cross-contamination. The purge-and-trap system may require extensive bake-out and cleaning after a high-level sample.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents.

3.5 Impurities in the purge gas and from organic compounds outgasing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic coating, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes: 10-uL, 25-uL, 100-uL, 250-uL, 500-uL, and 1,000 uL. These syringes should be equipped with a 20-gauge (0.006-in I.D.) needle having a length sufficient to extend from the sample inlet to within 1 cm of the glass frit in the purging device. The needle length will depend upon the dimensions of the purging device employed.

4.2 Syringe valve: Two-way, with Luer ends (three each), if applicable to the purging device.

4.3 Syringe: 5-mL, gas-tight with shutoff valve.

4.4 Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g.

4.5 Glass scintillation vials: 20-mL, with screw caps and Teflon liners or glass culture tubes with a screw cap and Teflon liner.

4.6 Volumetric flasks: 10-mL and 100-mL, Class A with ground-glass stoppers.

4.7 Vials: 2-mL, for GC autosampler.

4.8 Spatula: Stainless steel.

4.9 Disposable pipets: Pasteur.

4.10 Heater or heated oil bath: Should be capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C.

4.11 Purge-and-trap device: The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

4.11.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3-mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be utilized, provided equivalent performance is demonstrated.

4.11.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. It is recommended that 1.0 cm of methyl silicone-coated packing be inserted at the inlet to extend the life of the trap (see Figure 2). If it is not necessary to analyze for dichlorodifluoromethane or other fluorocarbons of smiliar volatility, the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated

and the polymer increased to fill the entire trap. Before initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the room, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

4.11.3 The desorber should be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode. The desorber design illustrated in Figure 2 meets these criteria.

4.11.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as shown in Figures 3 and 4.

4.11.5 Trap Packing Materials:

4.11.5.1 2,6-Diphenylene oxide polymer: 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.11.5.2 Methyl silicone packing: OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.11.5.3 Silica gel: 35/60 mesh, Davison, grade 15 or equivalent.

4.11.5.4 Coconut charcoal: Prepare from Barnebey Cheney, CA-580-26 lot #M-2649 by crushing through 26 mesh screen.

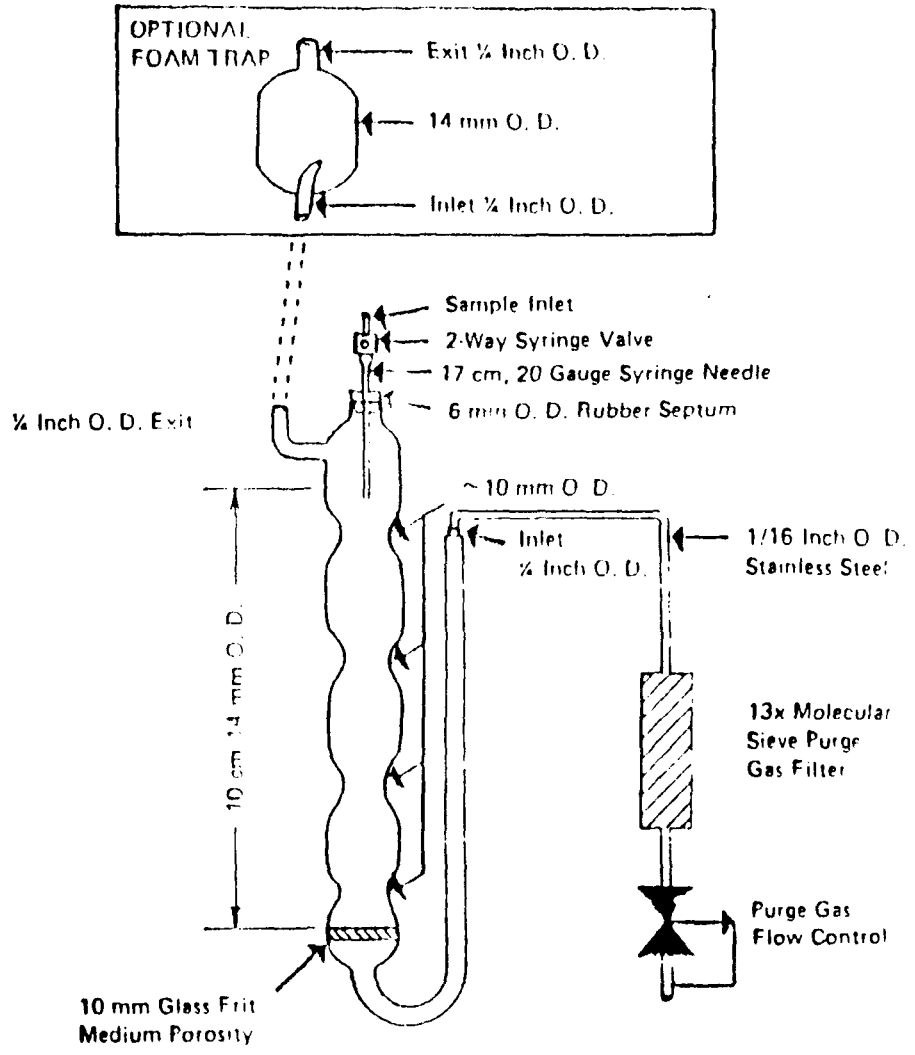


Figure 1. Purging chamber.

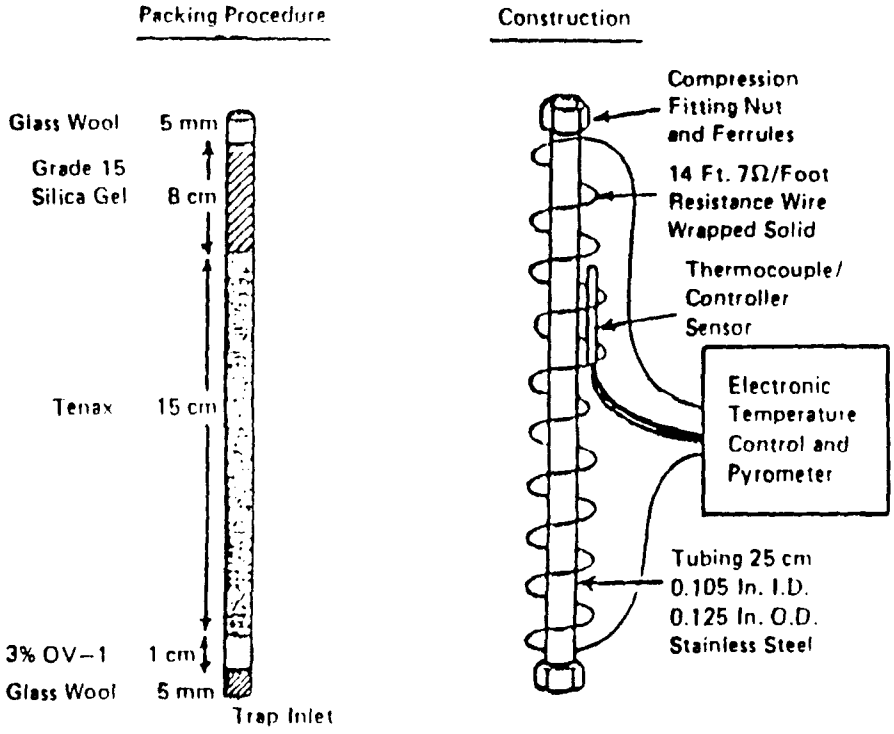


Figure 2. Trap packings and construction to include desorb capability for Method 8240.

4.12 Gas chromatograph/mass spectrometer system:

4.12.1 Gas chromatograph: An analytical system complete with a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

4.12.2 Column: 6-ft x 0.1-in. I.D. glass, packed with 1% SP-1000 on Carbopack-B (60/80 mesh) or equivalent.

4.12.3 Mass spectrometer: Capable of scanning from 35-260 amu every 3 sec or less, using 70 volts (nominal) electron energy in the electron impact mode and producing a mass spectrum that meets all the criteria in Table 3 when 50 ng of 4-bromofluorobenzene (BFB) are injected through the gas chromatograph inlet.

4.12.4 GC/MS interface: Any GC-to-MS interface that gives acceptable calibration points at 50 ng or less per injection for each of the analytes and achieves all acceptable performance criteria (see Table 3) may be used. GC-to-MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

4.12.5 Data system: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library should also be available.

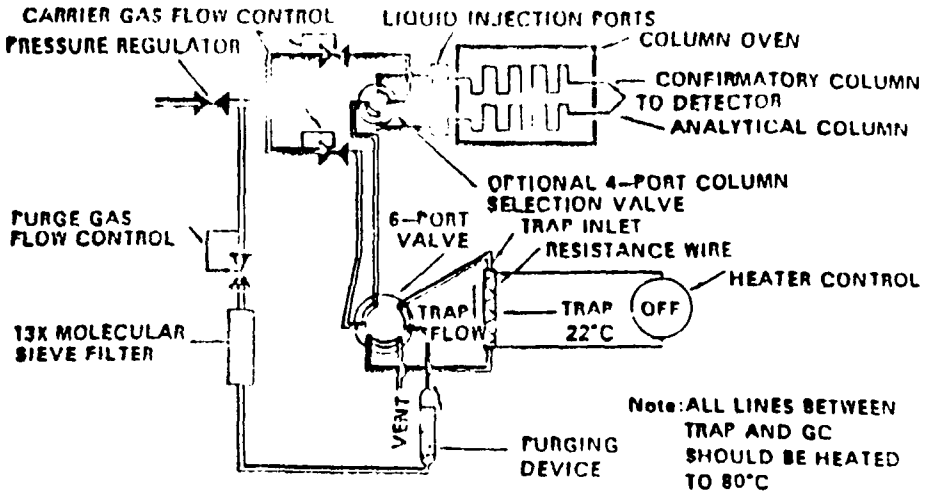


Figure 3. Schematic of purge-and-trap device – purge mode for Method 8240.

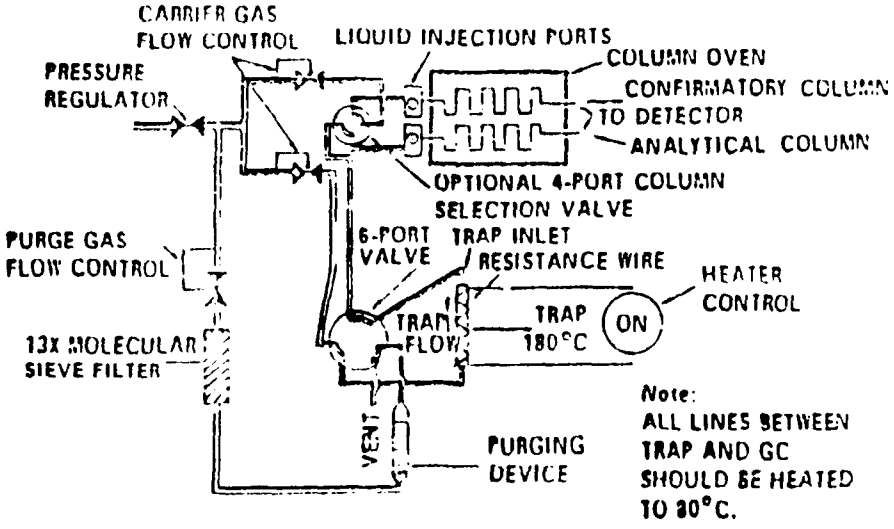


Figure 4. Schematic of purge-and-trap device – desorb mode for Method 8240.

TABLE 3. BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

5.0 REAGENTS

5.1 Stock solutions: Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.1.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.1.2 Add the assayed reference material, as described below.

5.1.2.1 Liquids: Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.1.2.2 Gases: To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side-arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.1.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.1.4 Transfer the stock standard solution into a Teflon-sealed screw cap bottle. Store, with minimal headspace, at -10°C to -20°C and protect from light.

5.1.5 Prepare fresh standards every two months for gases. Reactive compounds such as 2-chloroethylvinyl ether and styrene may need to be prepared more frequently. All other standards must be replaced after six months, or sooner if comparison with check standards indicates a problem.

5.2 Secondary dilution standards: Using stock standard solutions, prepare in methanol secondary dilution standards containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.3 Surrogate standards: The surrogates recommended are toluene-d_8, 4-bromofluorobenzene, and 1,2-dichloroethane-d_4. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described in Section 5.1, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 250 ug/10 mL in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 uL of the surrogate spiking solution prior to analysis.

5.4 Internal standards: The recommended internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Sections 5.1 and 5.2. It is recommended that the secondary dilution standard should be prepared at a concentration of 25 ug/mL of each internal standard compound. Addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 ug/L.

5.5 4-Bromofluorobenzene (BFB) standard: A standard solution containing 25 ng/uL of BFB in methanol should be prepared.

5.6 Calibration standards: Calibration standards at a minimum of five concentration levels should be prepared from the secondary dilution of stock standards (see Sections 5.1 and 5.2). Prepare these solutions in reagent water. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method (e.g., some or all of the compounds listed in Table 1 may be included). Store for one week only in a vial with no headspace.

5.7 Matrix spiking standards: Matrix spiking standards should be prepared from volatile organic compounds which will be representative of the compounds being investigated. The suggested compounds are 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The standard should be prepared in methanol, with each compound present at a concentration of 250 ug/10.0 mL.

5.8 Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standards be stored at -10°C to -20°C in screw-cap amber bottles with Teflon liners.

5.9 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit (MDL) of the parameters of interest.

5.9.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.9.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

5.9.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for 1 hr. While it is still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

5.10 Methanol: Pesticide quality or equivalent. Store apart from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes.

7.0 PROCEDURE

7.1 Direct injection: In very limited applications (e.g., aqueous process wastes), direct injection of the sample into the GC/MS system with a 10 uL syringe may be appropriate. One such application is for verification of the alcohol content of an aqueous sample prior to determining if the sample is ignitable (Methods 1010 or 1020). In this case, it is suggested that direct injection be used. The detection limit is very high (approximately 10,000 ug/L); therefore, it is only permitted when concentrations in excess of 10,000 ug/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

7.2 Initial calibration for purge-and-trap procedure:

7.2.1 Recommended GC/MS operating conditions:

Electron energy:	70 volts (nominal).
Mass range:	35-260 amu.
Scan time:	To give 5 scans/peak but not to exceed 7 sec/scan.
Initial column temperature:	45°C.
Initial column holding time:	3 min.
Column temperature program:	8°C/min.
Final column temperature:	220°C.
Final column holding time:	15 min.
Injector temperature:	200-225°C.
Source temperature:	According to manufacturer's specifications.

Transfer line temperature:	250-300°C.
Carrier gas:	Hydrogen at 50 cm/sec or helium at 30 cm/sec.

7.2.2 Each GC/MS system must be hardware-tuned to meet the criteria in Table 3 for a 50-ng injection or purging of 4-bromofluorobenzene (2-uL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.2.3 Assemble a purge-and-trap device that meets the specification in Section 4.11. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily for 10 min while backflushing at 180°C with the column at 220°C.

7.2.4 Connect the purge-and-trap device to a gas chromatograph.

7.2.5 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device. Add 5.0 mL of reagent water to the purging device. The reagent water is added to the purging device using a 5-mL glass syringe fitted with a 15-cm 20-gauge needle. The needle is inserted through the sample inlet shown in Figure 1. The internal diameter of the 14-gauge needle that forms the sample inlet will permit insertion of the 20-gauge needle. Next, using a 10-uL or 25-uL microsyringe equipped with a long needle (Paragraph 4.1), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards (Paragraph 5.6). Add the aliquot of calibration solution directly to the reagent water in the purging device by inserting the needle through the sample inlet. When discharging the contents of the mirco-syringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water. Similarly, add 10 uL of the internal standard solution (Paragraph 5.4). Close the 2-way syringe valve at the sample inlet.

7.2.6 Carry out the purge-and-trap analysis procedure as described in Section 7.4.1.

7.2.7 Tabulate the area response of the characteristic ions (see Table 1) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured (Section 7.5.2). The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

7.2.8 The average RF must be calculated for each compound. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds typically have RFs of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.2.8.1 Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.

7.2.8.2 Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

7.2.8.3 Tetrachloroethane and 1,1-dichloroethane: These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.9 Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs).

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

where:

RSD = relative standard deviation.

$\bar{x}$ = mean of 5 initial RFs for a compound.

SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N - 1}}$$

The %RSD for each individual CCC should be less than 30 percent. This criterion must be met in order for the individual calibration to be valid. The CCCs are:

1,1-Dichloroethene,
Chloroform,
1,2-Dichloropropane,
Toluene,
Ethylbenzene, and
Vinyl chloride.

7.3 Daily GC/MS calibration:

7.3.1 Prior to the analysis of samples, inject or purge 50-ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hr shift.

7.3.2 The initial calibration curve (Section 7.2) for each compound of interest must be checked and verified once every 12 hr of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC (Paragraph 7.3.3) and CCC (Paragraph 7.3.4).

7.3.3 System Performance Check Compounds (SPCCs): A system performance check must be made each 12 hr. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum response factor for volatile SPCCs is 0.300 (0.250 for Bromoform). Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.3.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Paragraph 7.2.9 are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\overline{RF}_I - RF_C}{\overline{RF}_I} \times 100$$

where:

$\overline{RF}_I$ = average response factor from initial calibration.

RF_C = response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid. If the criterion is not met (>25% difference), for any one CCC, corrective action MUST be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins.

7.3.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any

internal standard changes by more than 30 sec from the last check calibration (12 hr), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor or two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

7.4 GC/MS analysis:

7.4.1 Water samples:

7.4.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Two screening techniques that can be used are: the headspace sampler (Method 3810) using a gas chromatograph (GC) equipped with a photo ionization detector (PID) in series with an electrolytic conductivity detector (ECD); and extraction of the sample with hexadecane and analysis of the extract on a GC with a FID and/or an ECD (Method 3820).

7.4.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.4.1.3 Set up the GC/MS system as outlined in Paragraph 7.2.1.

7.4.1.4 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 7.3) before analyzing samples.

7.4.1.5 Adjust the purge gas (helium) flow rate to 25-40 mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response (see Section 7.2.8).

7.4.1.6 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20-mL syringe would allow the use of only one syringe.

If a second analysis is needed from a syringe, it must be analyzed within 24 hr. Care must be taken to prevent air from leaking into the syringe.

7.4.1.7 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.4.1.7.1 Dilutions may be made in volumetric flasks (10 to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.4.1.7.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

7.4.1.7.3 Inject the proper aliquot of samples from the syringe prepared in Paragraph 7.4.1.6 into the flask. Aliquots of less than 1-mL are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.4.1.7.4 Fill a 5-mL syringe with the diluted sample as in Paragraph 7.4.1.6.

7.4.1.8 Add 10.0 uL of surrogate spiking solution (Paragraph 5.3) and 10 uL of internal standard spiking solution (Paragraph 5.4) through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 uL of the surrogate spiking solution to 5 mL of sample is equivalent to a concentration of 50 ug/L of each surrogate standard.

7.4.1.9 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

7.4.1.10 Close both valves and purge the sample for 11.0 ± 0.1 min at ambient temperature.

7.4.1.11 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program and GC/MS data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with inert gas between 20 and 60 mL/min for 4 min. If this rapid heating requirement cannot be met, the gas chromatographic column must be used as a secondary trap by cooling it to 30°C (or subambient, if problems persist) instead of the recommended initial program temperature of 45°C.

7.4.1.12 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5-mL flushes of reagent water (or methanol followed by reagent water) to avoid carryover of pollutant compounds into subsequent analyses.

7.4.1.13 After desorbing the sample for 4 min, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 sec; then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C. Trap temperatures up to 220°C may be employed; however, the higher temperature will shorten the useful life of the trap. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.4.1.14 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.4.1.15 For matrix spike analysis, add 10 uL of the matrix spike solution (Paragraph 5.7) to the 5 mL of sample purged. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike standard.

7.4.1.16 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Sections 7.5.1 and 7.5.2 for qualitative and quantitative analysis.

7.4.2 Water-miscible liquids:

7.4.2.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with reagent water.

7.4.2.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100-mL volumetric flask and diluting to volume with reagent water. Transfer immediately to a 5-mL gas-tight syringe.

7.4.2.3 Alternatively, prepare dilutions directly in a 5-mL syringe filled with reagent water by adding at least 20 uL, but not more than 100-uL of liquid sample. The sample is ready for addition of internal and surrogate standards.

7.4.3 Sediment/soil and waste samples: It is highly recommended that all samples of this type be screened prior to the purge-and-trap GC/MS analysis. The headspace method (Method 3810) or the hexadecane extraction and screening method (Method 3820) may be used for this purpose. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. Use the screening data to determine whether to use the low-level method (0.005-1 mg/kg) or the high-level method (>1 mg/kg).

7.4.3.1 Low-level method: This is designed for samples containing individual purgeable compounds of < 1 mg/kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-level method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples. See Figure 5 for an illustration of a low soils impinger.

7.4.3.1.1 Use a 5-g sample if the expected concentration is <0.1 mg/kg or a 1-g sample for expected concentrations between 0.1 and 1 mg/kg.

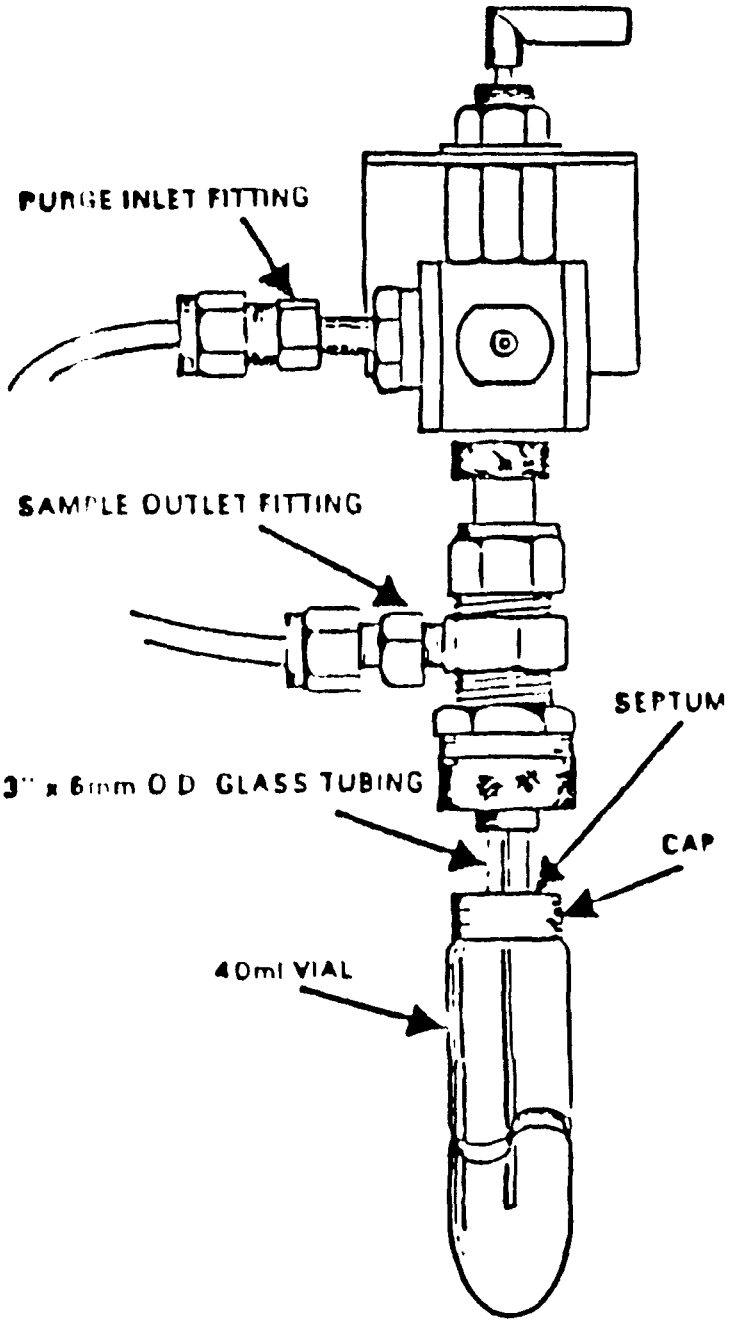


Figure 5. Low Soils Impinger

7.4.3.1.2 The GC/MS system should be set up as in Paragraphs 7.4.1.2-7.4.1.4. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature.

7.4.3.1.3 Remove the plunger from a 5-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of surrogate spiking solution (Paragraph 5.3) and internal standard solution (Paragraph 5.4) to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together.) The addition of 10 uL of the surrogate spiking solution to 5 g of sediment/soil is equivalent to 50 ug/kg of each surrogate standard.

7.4.3.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Paragraph 7.4.3.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.4.3.1.5 Determine the percent moisture of the soil/sediment sample. This includes waste samples that are amenable to moisture determination. Other wastes should be reported on a wet-weight basis. Immediately after weighing the sample, weigh (to 0.1 g) 5-10 g of additional sediment/soil into a tared crucible. Dry the contents of the crucibles overnight at 105°C. Allow to cool in a desiccator and reweigh the dried contents. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\% \text{ moisture} = \frac{\text{grams of sample - grams of dry sample}}{\text{grams of sample}} \times 100$$

7.4.3.1.6 Add the spiked reagent water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system. NOTE: Prior to the attachment of the purge device, the procedures in Paragraphs 7.4.3.1.4 and 7.4.3.1.6 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

7.4.3.1.7 Heat the sample to 40°C ± 1°C and purge the sample for 11.0 ± 0.1 min.

7.4.3.1.8 Proceed with the analysis as outlined in Paragraphs 7.4.1.11-7.4.1.16. Use 5 mL of the same reagent water as in the reagent blank. If saturated peaks occurred or would occur if a 1-g sample were analyzed, the medium-level method must be followed.

7.4.3.1.9 For low-level sediment/soils add 10 uL of the matrix spike solution (Paragraph 5.7) to the 5 mL of water (Paragraph 7.4.3.1.3). The concentration for a 5-g sample would be equivalent to 50 ug/kg of each matrix spike standard.

7.4.3.2 High-level method: The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to reagent water containing surrogate and internal standards. This is purged at ambient temperature. All samples with an expected concentration of > 1.0 mg/kg should be analyzed by this method.

7.4.3.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol weigh 4 g (wet weight) of sample into a tared 20-mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and determine the percent moisture of the sample using the procedure in Paragraph 7.4.3.1.5. For waste that is soluble in methanol, weigh 1 g (wet weight) into a tared scintillation vial or culture tube or a 10-mL volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 mL of methanol into the vial and mark the bottom of the meniscus. Discard this solvent.)

7.4.3.2.2 Quickly add 9.0 mL of methanol; then add 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 min. NOTE: Steps 7.4.3.2.1 and 7.4.3.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.4.3.2.3. Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 mL of reagent methanol to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at 4°C in the

dark, prior to analysis. The addition of a 100-uL aliquot of each of these extracts in Paragraph 7.4.3.2.6 will give a concentration equivalent to 6,200 ug/kg of each surrogate standard.

7.4.3.2.4 The GC/MS system should be set up as in Paragraphs 7.4.1.2-7.4.1.4. This should be done prior to the addition of the methanol extract to reagent water.

7.4.3.2.5 Table 4 can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. If a screening procedure was followed (Method 3810 or 3820), use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-level analysis to determine the appropriate volume. If the sample was submitted as a medium-level sample, start with 100 uL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.4.3.2.6 Remove the plunger from a 5.0-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10 uL of internal standard solution. Also add the volume of methanol extract determined in Paragraph 7.4.3.2.5 and a volume of methanol solvent to total 100 uL (excluding methanol in standards).

7.4.3.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

7.4.3.2.8 Proceed with the analysis as outlined in Paragraphs 7.4.1.11-7.4.1.16. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100 uL of methanol to simulate the sample conditions.

7.4.3.2.9 For a matrix spike in the medium-level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution (Paragraph 5.3), and 1.0 mL of matrix spike solution (Paragraph 5.7) as in Paragraph 7.4.3.2.2. This results in a 6,200 ug/kg concentration of each matrix spike standard when added to a 4-g sample. Add a 100-uL aliquot of this extract to 5 mL of water for purging (as per Paragraph 7.4.3.2.6).

TABLE 4. QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF MEDIUM-LEVEL SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
500-10,000 ug/kg	100 uL
1,000-20,000 ug/kg	50 uL
5,000-100,000 ug/kg	10 uL
25,000-500,000 ug/kg	100 uL of 1/50 dilution [b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a]The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

[b]Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

7.5 Data interpretation:

7.5.1 Qualitative analysis:

7.5.1.1 An analyte (e.g., those listed in Table 1) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference should be obtained on the user's GC/MS within the same 12 hours as the sample analysis. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of sample components at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

7.5.1.1.1 The sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hr as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

7.5.1.1.2 (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100% must be present in the sample spectrum). (2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent.

7.5.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

7.5.2 Quantitative analysis:

7.5.2.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (e.g., see Table 5).

7.5.2.2 Calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste:

$$\text{concentration (ug/L)} = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured.

I_s = Amount of internal standard injected (ng).

A_{is} = Area of characteristic ion for the internal standard.

RF = Response factor for compound being measured (Paragraph 7.2.7).

V_o = Volume of water purged (mL), taking into consideration any dilutions made.

TABLE 5. VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Acetone	Benzene	Bromofluorobenzene (surrogate)
Acrolein	Bromodichloromethane	Chlorobenzene
Acrylonitrile	Bromoform	Ethylbenzene
Bromomethane	2-Butanone	Ethyl methacrylate
Carbon disulfide	Carbon tetrachloride	2-Hexanone
Chloroethane	Chlorodibromomethane	4-Methyl-2-pentanone
Chloroform	2-Chloroethyl vinyl ether	Styrene
Chloromethane	Dibromomethane	1,1,2,2-Tetrachloroethane
Dichlorodifluoromethane	1,4-Dichloro-2-butene	Tetrachloroethene
1,1-Dichloroethane	1,2-Dichloropropane	Toluene
1,2-Dichloroethane	cis-1,3-Dichloropropene	Toluene-d_8 (surrogate)
1,2-Dichloroethane-d_4 (surrogate)	trans-1,3-Dichloropropene	1,2,3-Trichloropropane
1,1-Dichloroethene	1,1,1-Trichloroethane	Xylene
trans-1,2-Dichloroethene	1,1,2-Trichloroethane	
Iodomethane	Trichloroethene	
Methylene chloride	Vinyl acetate	
Trichlorofluoromethane		
Vinyl chloride		

Sediment/Soil, Sludge, and Waste:

High-level:

$$\text{concentration (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)}$$

Low-level:

$$\text{concentration (ug/kg)} = \frac{(A_x)(I_s)}{(A_{is})(RF)(W_s)}$$

where:

A_x, I_s, A_{is}, RF = same as for water.

V_t = volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made).

V_i = volume of extract added (uL) for purging.

W_s = weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

7.5.2.3 Sediment/soil samples are generally reported on a dry weight basis, while sludges and wastes are reported on a wet weight basis. The % moisture of the sample (as calculated in Paragraph 7.4.3.1.5) should be reported along with the data in either instance.

7.5.2.4 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulas given above should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.5.2.5 Report results without correction for recovery data. When duplicates and spiked samples are analyzed, report all data obtained with the sample results.

8.0 QUALITY CONTROL

8.1 Each laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of

analyses meet the performance characteristics of the method. When results of sample spikes indicate a typical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst should demonstrate, through the analysis of a reagent water blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent water blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

8.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still useable, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., column changed), recalibration of the system must take place.

8.4 Required instrument QC is found in the following section:

8.4.1 The GC/MS system must be tuned to meet the BFB specifications in Section 7.2.2.

8.4.2 There must be an initial calibration of the GC/MS system as specified in 7.2.

8.4.3 The GC/MS system must meet the SPCC criteria specified in 7.3.3 and the CCC criteria in 7.3.4, each 12 hr.

8.5 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.5.1 A quality (QC) check sample concentrate is required containing each analyte at a concentration of 10 ug/mL in methanol. The QC check sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC check sample concentrate must be made using stock standards prepared independently from those used for calibration.

8.5.2 Prepare a QC check sample to contain 20 ug/L of each analyte by adding 200 uL of QC check sample concentrate to 100 mL of reagent water.

8.5.3 Four 5-mL aliquots of the well-mixed QC check sample are analyzed according to the method beginning in Section 7.4.1.

8.5.4 Calculate the average recovery ($\bar{x}$) in ug/L, and the standard deviation of the recovery (s) in ug/L, for each analyte using the four results.

8.5.5 For each analyte compare s and $\bar{x}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and $\bar{x}$ for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{x}$ falls outside the range for accuracy, then the system performance is unacceptable for that analyte. NOTE: The large number of analytes in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined.

8.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Paragraph 8.5.6.1 or 8.5.6.2.

8.5.6.1 Locate and correct the source of the problem and repeat the test for all analytes beginning with Section 8.5.2.

8.5.6.2 Beginning with Section 8.5.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.5.2.

8.6 The laboratory must, on an ongoing basis, analyze a reagent blank, a matrix spike, and a matrix spike duplicate/duplicate for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.6.1 The concentration of the spike in the sample should be determined as follows:

8.6.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.6.2, whichever concentration would be larger.

8.6.1.2 If the concentration of a specific analyte in the sample is not being check against a specific limit, the spike should be at 20 ug/L or 1 to 5 times higher than the background concentration determined in Section 8.6.2, whichever concentration would be larger.

TABLE 6. CALIBRATION AND QC ACCEPTANCE CRITERIA[a]

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for X (ug/L)	Range p, p_s (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.4	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.4	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethyl benzene	11.8-28.2	7.5	17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.5-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Q = Concentration measured in QC check sample, in ug/L.
s = Standard deviation of four recovery measurements, in ug/L.
X = Average recovery for four recovery measurements, in ug/L.
p, p_s = Percent recovery measured.
D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

8.6.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each analyte. If necessary, prepare a new QC check sample concentrate (Section 8.5.1) appropriate for the background concentration in the sample. Spike a second 5-mL sample aliquot with 10 uL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as 100(A-B)%/T, where T is the known true value of the spike.

8.6.3 Compare the percent recovery (p) for each analyte with the corresponding QC acceptance criteria found in Table 6. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1. If spiking was performed at a concentration lower than 20 ug/L, the analyst must use either the QC acceptance criteria presented in Table 6, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of an analyte: (1) Calculate accuracy (x') using the equation found in Table 7, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 7, substituting x' for $\bar{x}$; (3) calculate the range for recovery at the spike concentration as (100x'/T) ± 2.44(100S'/T)%.

8.6.4 If any individual p falls outside the designated range for recovery, that analyte has failed the acceptance criteria. A check standard containing each analyte that failed the criteria must be analyzed as described in Section 8.7.

8.7 If any analyte fails the acceptance criteria for recovery in Section 8.6, a QC check standard containing each analyte that failed must be prepared and analyzed. NOTE: The frequency for the required analysis of a QC check standard will depend on the number of analytes being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of analytes in Table 6 must be measured in the sample in Section 8.6, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

8.7.1 Prepare the QC check standard by adding 10 uL of the QC check sample concentrate (Section 8.5.1 or 8.6.2) to 5 mL of reagent water. The QC check standard needs only to contain the analytes that failed criteria in the test in Section 8.6.

8.7.2 Analyze the QC check standard to determine the concentration measured (A) of each analyte. Calculate each percent recovery (p_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.7.3 Compare the percent recovery (p_s) for each analyte with the corresponding QC acceptance criteria found in Table 6. Only analytes that failed the test in Section 8.6 need to be compared with these criteria. If the recovery of any such analyte falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem must be immediately identified and corrected. The result for that analyte in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.8 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of five spiked samples (of the same matrix) as in Section 8.6, calculate the average percent recovery ($\bar{p}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{p} - 2s_p$ to $\bar{p} + 2s_p$. If $\bar{p} = 90\%$ and $s_p = 10\%$, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis (e.g., after each five to ten new accuracy measurements).

8.9 To determine acceptable accuracy and precision limits for surrogate standards the following procedure should be performed.

8.9.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

8.9.2 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (p) and standard deviation of the percent recovery (s) for each of the surrogates.

8.9.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

8.9.4 For aqueous and soil matrices, these laboratory established surrogate control limits should, if applicable, be compared with the control limits listed in Table 8. The limits given in Table 8 are multi-laboratory performance based limits for soil and aqueous samples, and therefore, the single-laboratory limits established in Paragraph 8.9.3 must fall within those given in Table 8 for these matrices.

8.9.5 If recovery is not within limits, the following procedures are required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

TABLE 7. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Parameter	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s_r' (ug/L)	Overall precision, S' (ug/L)
Benzene	0.93C+2.00	0.26X-1.74	0.25X-1.33
Bromodichloromethane	1.03C-1.58	0.15X+0.59	0.20X+1.13
Bromoform	1.18C-2.35	0.12X+0.34	0.17X+1.38
Bromomethane	1.00C	0.43X	0.58X
Carbon tetrachloride	1.10C-1.68	0.12X+0.25	0.11X+0.37
Chlorobenzene	0.98C+2.28	0.16X-0.09	0.26X-1.92
Chloroethane	1.18C+0.81	0.14X+2.78	0.29X+1.75
2-Chloroethylvinyl ether[a]	1.00C	0.62X	0.84X
Chloroform	0.93C+0.33	0.16X+0.22	0.18X+0.16
Chloromethane	1.03C-1.81	0.37X+2.14	0.58X+0.43
Dibromochloromethane	1.01C-0.03	0.17X-0.18	0.17X+0.49
1,2-Dichlorobenzene[b]	0.94C+4.47	0.22X-1.45	0.30X-1.20
1,3-Dichlorobenzene	1.06C+1.68	0.14X-0.48	0.18X-0.82
1,4-Dichlorobenzene[b]	0.94C+4.47	0.22X-1.45	0.30X-1.20
1,1-Dichloroethane	1.05C+0.36	0.13X-0.05	0.16X+0.47
1,2-Dichloroethane	1.02C+0.45	0.17X-0.32	0.21X-0.38
1,1-Dichloroethene	1.12C+0.61	0.17X+1.06	0.43X-0.22
trans-1,2,-Dichloroethene	1.05C+0.03	0.14X+0.09	0.19X+0.17
1,2-Dichloropropane[a]	1.00C	0.33X	0.45X
cis-1,3-Dichloropropene[a]	1.00C	0.38X	0.52X
trans-1,3-Dichloropropene[a]	1.00C	0.25X	0.34X
Ethyl benzene	0.98C+2.48	0.14X+1.00	0.26X-1.72
Methylene chloride	0.87C+1.88	0.15X+1.07	0.32X+4.00
1,1,2,2-Tetrachloroethane	0.93C+1.76	0.16X+0.69	0.20X+0.41
Tetrachloroethene	1.06C+0.60	0.13X-0.18	0.16X-0.45
Toluene	0.98C+2.03	0.15X-0.71	0.22X-1.71
1,1,1-Trichloroethane	1.06C+0.73	0.12X-0.15	0.21X-0.39
1,1,2-Trichloroethane	0.95C+1.71	0.14X+0.02	0.18X+0.00
Trichloroethene	1.04C+2.27	0.13X+0.36	0.12X+0.59
Trichlorofluoromethane	0.99C+0.39	0.33X-1.48	0.34X-0.39
Vinyl chloride	1.00C	0.48X	0.65X

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.
s_r' = Expected single analyst standard deviation of measurements at an average concentration of X, in ug/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of X, in ug/L.
C = True value for the concentration, in ug/L.
X = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

[a]Estimates based upon the performance in a single laboratory.
[b]Due to chromatographic resolution problems, performance statements for these isomers are based upon the sums of their concentrations.

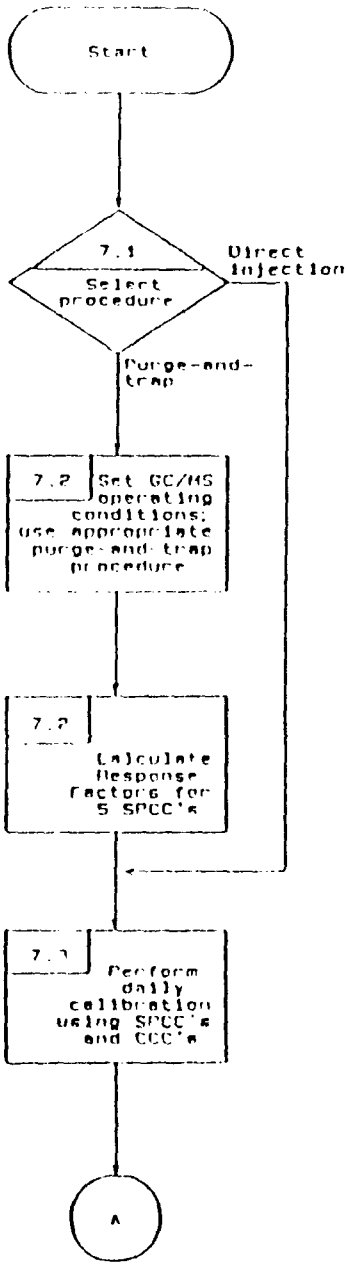
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

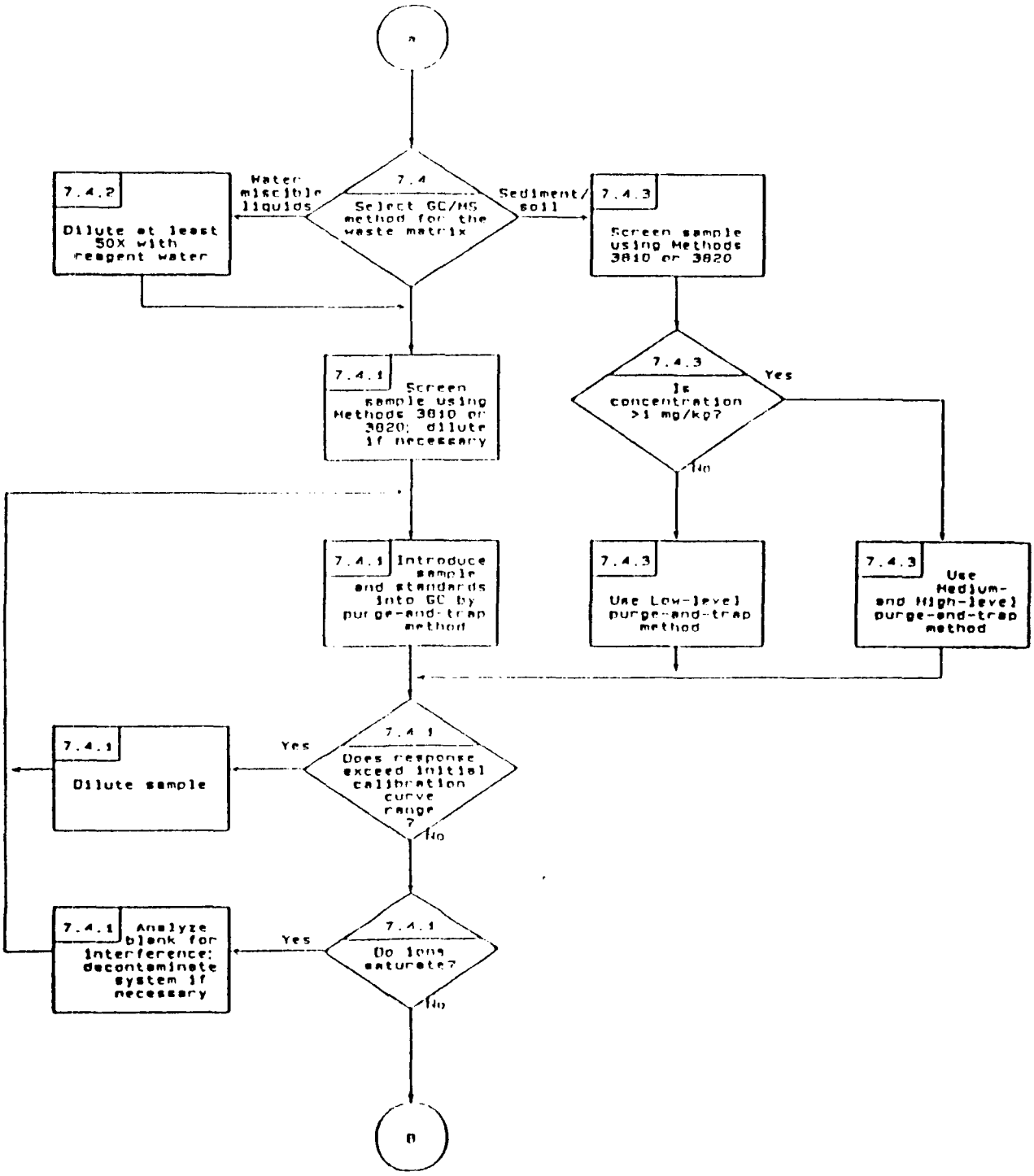
8.9.6 At a minimum, each laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

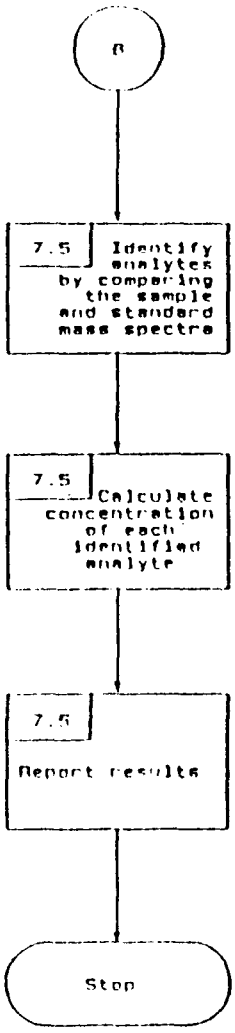
8.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column or a different ionization mode using a mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

TABLE 8. SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Low/Medium Water	Low/Medium Soil/Sediment
4-Bromofluorobenzene	86-115	74-121
1,2-Dichloroethane-d_4	76-114	70-121
Toluene-d_8	88-110	81-117


Start
7.1
Select procedure
Direct injection
Purge-and-trap
7.2
Set GC/MS operating conditions; use appropriate purge-and-trap procedure
7.2
Calculate Response Factors for 5 SPCC's
7.3
Perform daily calibration using SPCC's and CCC's
A


A
7.4
Select GC/MS method for the waste matrix
Water miscible liquids
Sediment/ soil
7.4.2
Dilute at least 50X with reagent water
7.4.3
Screen sample using Methods 3810 or 3820
7.4.3
Is concentration >1 mg/kg?
Yes
No
7.4.1
Screen sample using Methods 3810 or 3820; dilute if necessary
7.4.1
Introduce sample and standards into GC by purge-and-trap method
7.4.3
Use Low-level purge-and-trap method
7.4.3
Use Medium- and High-level purge-and-trap method
7.4.1
Does response exceed initial calibration curve range ?
Yes
No
7.4.1
Dilute sample
7.4.1
Do ions saturate?
Yes
No
7.4.1
Analyze blank for interference; decontaminate system if necessary
B


B
7.5
Identify analytes by comparing the sample and standard mass spectra
7.5
Calculate concentration of each identified analyte
7.5
Report results
Stop

ALS/ANALYTICAL LABORATORY SERVICES

DIVISION OF M C CHEMICAL CO.

QUALITY ASSURANCE & QUALITY CONTROL MANUAL

720 South St.
Rockford, Il. 61102

1989

PERFORMANCE AND SYSTEM AUDIT:

The QA Officer (to be named) will carry out performance and systems audits to ensure that data of known and defensible quality are produced during the program.

Systems audits are qualitative evaluation of all components of field and laboratory quality control measurement systems. They determine if the measurement systems are being used right.

The performance audit is a quantitative evaluation of the measurement systems of a program. It requires testing the measurement systems with samples of known composition or behavior to evaluate precision and accuracy.

INTRODUCTION:

Quality assurance program has two primary functions in the laboratory. First the program should continually monitor the reliability (accuracy and precision) of the results reported. This function is the determination of quality. The second function is the control of quality (to meet the program requirements for reliability).

Each analytical method has a rigid protocol. Similarly, Quality Control associated with a test must include definite required steps for monitoring the test, and ensuring that the tests results are correct. The steps in Quality Control vary with the type of analysis.

This handbook discusses the basic factors of Quality Control in ALS/Analytical Laboratory Services. Laboratory measurements determine the value of analytical results and provide recommendations for the control of these factors to ensure that analytical results are the best possible. Quality Assurance programs initiate from and based upon these recommendations should increase confidence in the reliability of the reported analytical results.

This manual describes the Quality Assurance Program in use at ALS/Analytical Laboratory Services, a division of M C Chemical Rockford, Illinois. It is intended for review by ALS clients, potential clients and internally as a reference for company personnel.

PURPOSE AND SCOPE:

The purpose of quality control is to monitor and control the generation of data. ALS program ensures that correct methodology, proper calibration and systematic quality control procedures are used. This manual describes in detail the analytical methods, laboratory services, instrumentation, glassware, reagents, analytical performance, sample handling, data processing and reporting, safety procedures, etc., which are all part of a comprehensive quality control program.

ANALYTICAL METHODS:

Most of the analytical methods used by ALS/Analytical Laboratory Services have been documented by the Environmental Protection Agency (EPA) as approved methodologies.

A list of sources employed may be found in the Reference Section. Methodologies used for specific analysis are found on Table 1.

The methods which are routinely used are contained in a ALS/ Analytical Laboratory Services Methods Manual. As new methods are accepted, they are reviewed, changed as necessary and become part of the approved methods. The manual is reviewed and upgraded on an annual basis.

SAMPLE HANDLING, DATA PROCESSING AND REPORTING OF RESULTS:

Each sample entering the laboratory is assigned a number. This number is affixed to the sample container and recorded into a bonded sample log book. The sample number, client, date received, client's sample identification and parameters requested are entered into the log book. A laboratory data sheet is completed for each group of samples received and accompanies the samples to the laboratory. ALS data sheet shown in Figure 1.

The sample is split and preserved as required (Table 2). Samples with short holding times are delivered immediately to the designated analyst. All samples, except metals are stored at 4°C until testing is completed, at which time, any remaining sample is removed to an ambient storage and retained for a minimum of thirty days.

Analyst record their work in a permanent record book. There is a book provided for each parameter tested by the laboratory.

As the requested testing is completed, the analytical results are compiled on the Laboratory Data Sheet and routed to the Quality Controller. The report is reviewed for calculation errors, proper significant figures and detection limits.

CHAIN OF CUSTODY PROCEDURE:

The purpose is to provide an accurate written record which can be used to trace the possession and handling of the samples from collection through analysis and introduction as evidence.

A sample is in someone's custody if:

1. It is in one's actual physical possession, or
2. It is in one's view after being in one's physical possession, or
3. It is in one's physical possession and then locked up so that no one can tamper with it, or
4. It is kept in a secured area restricted to authorized personnel only.

SAMPLE COLLECTION, HANDLING, AND IDENTIFICATION:

A minimum number of people will be involved in sample collection and handling. In most instances this will include the sample collector(s sample custodian and the analyst(s).

A Chain of Custody Form will be completed at the time of sample collection by the sample collector(s).

All records and labels (Figure 3) will be filled out legibly, in ink.

TRANSFER OF CUSTODY:

Samples and record will be kept in custody as described above. When transferring the possession of the sample(s), the transferee must sign and record the date and time on the Chain of Custody record form (Figure 4).

All samples sent to the laboratory must be accompanied by the Chain of Custody record and other pertinent forms. A copy of the forms should be retained by the originator. (Photocopy or carbon)

Shipping receipts are retained as part of the permanent Chain of Custody documentation.

LABORATORY CUSTODY PROCEDURE:

Laboratory Director is the designated Chain of Custody Custodian, an alternate to be named later. All incoming samples are received by the custodian who indicates receipt by signing the accompanying custody forms and retains the signed forms as permanent records.

Permanent logbooks are maintained by the custodian for each and every sample.

All chain of custody records and samples are kept under lock, and the keys are kept by the Chain of Custody Custodian. The custodian shall see that samples are properly stored and maintained prior to analysis.

Distribution of samples to the analysts will be made only by the custodian. To remove samples from storage, the analyst must sign for the samples on the Chain of Custody Form, and samples must be returned to the custodian, to secured storage at the end of each day or when analysis are completed.

All analytical work is recorded in Chain of Custody log books. The books are distributed to the analyst(s) by the custodian. The log books must be returned to the custodian and to the secured file at the end of each day or when analysis are completed. The unused portion of samples will be retained under custody until destroyed or returned to the client. All laboratory records will be retained in the locked Chain of Custody file.

LABORATORY SECURITY:

All laboratory doors will be locked to assure access to ALS employees only. Also the laboratory is monitored by a (MC employee) Alarm Security System.

MONITORING ANALYTICAL PERFORMANCE:

All routinely performed tests are subject to initial evaluation of precision and accuracy. In addition, daily performance monitoring of precision and accuracy is done to assure conformance to acceptable levels.

The design of the daily Quality Control includes:

1. Blanks and appropriate standards are analyzed with each set of samples.
2. Ten percent of the samples are run in duplicate for the documentation of precision.
3. Ten percent of the samples are spiked to document the recovery and apparent accuracy of the method.

4. Where applicable, stable control samples of known content are analyzed with each group of samples.
5. Blind samples are submitted to the laboratory without the analyst having knowledge of their origin.

The data gathered from these routine evaluations is used to calculate and maintain both precision (R) and accuracy (P) control limits. These tabulators provide the data rejection criteria for normal laboratory operations. Should the results of replicate or spiked analysis fal outside control limits, the system is thoroughly evaluated, and corrective action taken. The suspected data is rejected and samples are reanalyzed prior to reporting. The data tabulations are valuable not only because they provide a current status report on the quality of results, but because they are an excellent troubleshooting tool in alerting the laboratory to trends in accuracy and precision of data before out of control situations occur. In addition they also provide documentation of the levels of precision and accuracy at any point in time should the validity of analysts be questioned at a later date.

It is the responsibility of the analysts and laboratory supervisor to see that prescribed Quality Control is done and duly recorded.

Recent Quality Control Records for all parameters, regularly determined in ALS Laboratory are in designated ring binders and are kept in the file in the laboratory.

Older records are in a file storage box in the storage area.

CALIBRATION AND MAINTENANCE:

Calibration is the process by which a standard or piece of equipment of a given accuracy is compared against a standard or piece of equipment of a higher accuracy. Adjustments are made as necessary to assure that the standards of equipment are within the prescribed accuracy Preventive maintenance is an orderly program of positive actions (equipment cleaning, lubricating, adjusting, reconditioning,and/or testing) to prevent instruments from failing during use.

INSTRUMENTATION AND CALIBRATION:

ALS/Analytical Laboratory Services maintains all instruments and major equipment on specified calibration schedules.

Oven temperatures are checked at the beginning and end of each day of use. If a trend in inaccuracy is found which cannot be corrected by ALS personnel, professional service is obtained.

Analytical balances are checked with class S weights at least monthly. If inaccuracies are found that cannot be corrected internally, professional services are obtained. The balances are serviced and checke by an NBS certified service agent each year.

The pH meter is calibrated every morning by a two buffer calibration mode and also with a single buffer before every sample.

The Parr 1261 Isoperibol Calorimeter will be calibrated yearly to get an accurate mean value. Single standardizing tests will be performed monthly as a check that no part of the apparatus has been damaged or has altered its characteristics.

SpectrAA Atomic Absorption Spectrometer; Atomic Absorption Spectrophotometers are calibrated for each metal analyzed and a record is kept of instrument response. Should a lack of sensitivity or other malfunction be detected that cannot be corrected in-house, professional service is obtained.

Gas chromatographs are calibrated before use for sensitivity and accuracy. The standing current of electron capture detectors are recorded before each days use. If the required sensitivity cannot be obtained, prescribed maintenance procedures are initiated. If instrument response is not enhanced to the desired level, professional service is obtained.

The Oxygen demand meter is calibrated on a regular basis with the saturated air method.

The Conductivity meter is calibrated daily by a KCl standard.

PROCUREMENT CONTROL:

All chemicals ordered will be reagent grade or ACS-grade. The chemicals are ordered by the laboratory supervisor in quantities sufficient to accommodate the laboratory procedures. When the reagent is received, the chemical will be labeled with (Label A) and the correct information being filled out. The laboratory technician will also record the chemical in the chemical receiving log (date received, date expired, chemical name, and hazard class). The chemicals will then be put in storage. All reagents will be discarded after 9 months unless otherwise specified.

Chemicals, Solvents and Gases; The purity is specified for all chemical reagents, solvents and gases used in ALS Laboratory. ACS analytical reagent grade chemicals are specified for most test methods.

Standard solutions are stored in borosilicate glass bottles or polyethylene containers, which ever is appropriate. Containers are dated and initialed by the preparer. When prescribed shelf-lives are reached, solutions are discarded and freshly prepared.

All organic and inorganic chemicals are dated upon arrival at the laboratory to monitor shelf life.

Fuels are commercial grade:

A. High purity grade nitrogen
B. High purity argon
C. High purity acetylene
D. High purity nitrous oxide
E. High purity helium
F. High purity hydrogen
G. Highly purified Air (Oilless Compressor)

Water; There are two grades of reagent water used in the laboratory. The tap water used in the laboratory is Rockford City Water. Its primary use is for the washing of glassware and sample containers. The water is checked daily to determine that the proper quality is being maintained. A permanent record of the specific conductance of the deionized water is recorded daily. See Table 3 requirements for Reagents Water.

PREPARATION OF 0.1M HYDROCHLORIC ACID:

Fill 1 litre ground-glass stoppered bottle with almost 1 litre of distilled water. Add about 9mL of concentrated HCL from a measuring pipet or graduated cylinder and mix well.

2. Add 20mL of the iodine solution to be standardized and dilute to 300mL with type II water.
3. Titrate with 0.0250N sodium thiosulfate until pale straw color.
4. Add small amount of starch indicator and wait until a homogeneous blue color develops.
5. Continue titration, drop by drop, until the color disappears.
6. Run in duplicates.
7. Calculate normality by:

$$(N)I_2 = \frac{ML\ NaS_2O_3 \times 0.0250}{20}$$

SAFETY:

It is dangerous to assume that personnel at any level of work have adequate information about laboratory safety.

Each employee is expected to familiarize herself/himself with the safety rules and regulations and is responsible for safety on the job for himself and fellow workers.

Management is responsible for providing safe operating conditions.

SAFETY PROCEDURES:

1. Laboratory Conduct;
   a) Follow instructions EXACTLY
   b) Perform only authorized experiments
   c) Protect eyes, face, hands, and body
   d) Practice good housekeeping
   e) Learn basic first aid
   f) Know where to get help quickly
   g) Know location of first aid and fire fighting equipment
   h) Report all accidents and unusual occurrences immediately
   i) Be professional
2. Personnel Safety;
   a) Clean up all water spills on the floor
   b) Dispose of broken glass only in marked containers
   c) Wear appropriate clothing; a rubber apron
3. Fire Prevention;
   a) Store all flammable liquids in a fireproof cabinet
   b) Whenever possible, and always when large quantities are involved, use flammable liquids in a fume hood. When it is necessary to use flammables on an open bench, be certain that there are no flames nearby.
   c) Place waste flammable liquids in the appropriate safety cans for disposal.
   d) Dispose of solid and liquid oxidants appropriately.
   e) Be certain not to overload electrical circuits
   f) Do not use equipment with worn or bare wires
   g) Be aware of the types of fire extinguishers in the laboratory
   h) Know the location of the fire extinguishers

4. Prevention Of Poisoning;
   a) Use toxic materials such as chlorine gas, cyanides and bromine in a hood only. These are inhalation hazards and some are toxic by skin absorption.
   b) Use gloves when handling bromine
   c) Some compounds in use in the laboratory are slow-acting poisons when ingested or absorbed in small amounts. Among these are arsenic, mercury, lead and hexavalent chromium compounds. Wear gloves when handling these compounds in high concentrations, and wash hands thoroughly after use.
   d) Clean up all chemical spills, even if seemingly harmless materials. One spill may react with another. Neutralize concentrated acids with sodium carbonate (Na_2CO_3) and bases with boric acid H_3BO_4 before cleaning up.
   e) Always use a rubber bulb to pipet.
   f) Exercise care in handling of all samples when their contents are unknown or known.
   g) Do not eat, drink or smoke in the laboratory work areas.
   h) If you have any questions about handling a particular compound or reaction, consult references.
5. Safety Equipment;
   a) Know how to use the following items in the laboratory.
      I. Fire extinguishers
      II. Eye wash stations
      III. First aid kits
   b) General;
      I. Protective clothing
      II. Eye protection
      III. Foot protection
      IV. Miscellaneous: Rubber, cloth or leather gloves are available for hand protection, and must be worn whenever warranted.
      V. Exits: Be conscious at all times of the nearest laboratory exit and nearest building exit.
      VI. Smoking: Prohibited
      VII. Food: Do not eat or drink in the laboratory, or store lunches in refrigerators used for chemicals

DEFINITIONS:

1. Accuracy-Means the nearness of a result or the mean (X) of a set of results to the true value. Accuracy is assessed by means of reference samples and percent recoveries.

2. Analytical Batch-The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition.

3. Blank-A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix: However a universal blank matrix does not exist for solid samples and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

   a. Reagent Blank-Is an aliquot of analyte-free water or solvent analyzed with the analytical batch.
   b. Field Blanks-Are aliquots of analyte-free water or solvents brought to the field in sealed containers and transported back to the laboratory with the sample containers.
   c. Trip Blanks-Are not opened in the field. These blanks check on sample contamination originating from sample transport, shipping and from site conditions.
   d. Equipment Blanks-Are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, and returned to the laboratory as a sample. Also are a check on sampling device cleanliness.

4. Calibration Check-Verification of the ratio of instrument response to analyte amount. It is done by analyzing for analyte standard in an appropriate solvent. They are made from a stock solution which is different from the stock used to prepare standards.

5. Check Sample-A blank which has been spiked with the analyte from an independent source in order to monitor the execution of the analytical method. The level of the spike shall be at the regulatory action level when applicable. Otherwise, the spike shall be at 5 times the estimate of the quantification unit. The matrix used shall be phase matched with the samples and well characterized: For example-reagent water is appropriate for an aqueous sample.

6. Environmental Sample-Is a representative sample of any material (aqueous, nonaqueous or multimedia) collected from any source for which determination of composition or contamination is requested or required.

   a. Water/wastewater-Raw sources waters for public drinking water supplies, ground waters, municipal influents/effluents and industrial influents/effluents.

b. Sludge-Municipal sludges and industrial sludges.
c. Waste-Aqueous and nonaqueous liquid wastes, chemical solids, contaminated soils, and industrial liquid and solid wastes.

7. Matrix/Spike-Duplicate analysis: Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction/digestion and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level or the estimated or actual method quantification limit. When the concentration of the analyte in the sample is greater than 0.190, no spike of the analyte is necessary.

8. (MQL) The Method Quantification Limit-Is the minimum concentration of a substance that can be measured and reported.

9. Precision-Means the measurement of agreement of a set of replicate results among themselves without assumption of any prior information as to the true result. Precision is assessed by means of duplicate/replicate sample analysis.

10. (PQL) The Practical Quantification Limit-Is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

11. (RCRA) The Resource Conservation and Recovery Act.

12. Reagent Grade-Conform to the current specifications of the committee on analytical reagents of the American Chemical Society.

13. Replicate Sample-Is a sample prepared by dividing a sample into two or more separate aliquots.

14. Standard Curve-Is a curve which plots concentrations of know analyte standard versus the instrument response to the analyte

15. Surrogate-Are organic compounds which are similar to analyte of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

16. Water-Reagent, analyte-free, laboratory pure water, means distilled or deionized water or type II reagent water which is free of contaminants, that may interfere with the analytical test in question.

GLASSWARE CLEANING PROCEDURES

The cleaning method for glassware is dependent upon the use to which it will be put. General use glassware is washed in sink with HOT WATER and laboratory detergent. It receives a final distilled water rinse.

General laboratory glassware and general sample container:

1. Remove any foreign material in glassware or container with laboratory detergent solution, hot water and brush.

2. Rinse thoroughly at least twice or as many times as necessary with warm water, to remove all detergent.

3. Rinse thoroughly at least twice with deionized water.

4. If a grease film remains, use acetone to dissolve and remove the grease. Then follow steps 1, 2, & 3.

5. If any additional residue remains, rinse and/or soak the item with 1+1 hydrochloric acid (HCL) then follow the general washing procedure (steps 1, 2, & 3).

CORRECTIVE ACTION

1. Identification and definition of the problem.

2. Assignment of responsibility for investigating the problem.

3. Determination of a corrective action to eliminate the problem.

4. Assigning and accepting responsibility for implementing the corrective action.

5. Implementing the corrective action and evaluating its effectiveness.

6. Verify that the corrective action has eliminated the problem

All sets of corrective action will be documented on the standard correction action form CA-1.

ANALYTICAL QUALITY CONTROL

1. DUPLICATED SPIKE: Will be performed at least once with each analytical batch with a minimum of once per twenty samples.

2. BLANKS: Each batch shall be accompanied by a reagent blank. The reagent blank shall be carried through the entire analytical procedure.

Form
CA-1

CORRECTIVE ACTION

What is the problem? Identify and define -

_____

_____

_____

_____

NOTES: Investigating the problem.

Solve the problem; Corrective action.

Assigning and accepting responsibility.

Implementing corrective action.

Verify the corrective action worked.

DATE:_____ SIGNATURE:_____

US4650

SAMPLING - QUALITY ASSURANCE AND QUALITY CONTROL

QUALITY CONTROL:

1. Field Samples;

   a) Trip Blanks - Should accompany sample containers to and from the field. These samples can be used to detect any contamination or cross-contamination during handling and transportation.

   b) Field Blanks - Should be collected at specified frequencies, which will vary according to the probability of contamination or cross-contamination. Field blanks are often metal and/or organic free water aliquots that contact sampling equipment under field conditions and are analyzed to detect any contamination from sampling equipment, cross contamination from previously collected samples, or contamination from condition during sampling (eg airborne contaminants that are not from the waste being sampled).

   c) Field Duplicates - Field duplicates are collected at specified frequencies and are employed to documents precision. The precision resulting from field duplicates is a function of the variance of waste composition, the variance of the sampling technique, and the variance of the analytical technique.

   d) Field Spikes - Are infrequently used to determine the loss of parameters of interest during sampling and shipments to the laboratories. Because spiking is done in the field, the making of spiked samples or spiked blanks is susceptible to error. In addition, compounds can be lost during spiking, and equipment can be contaminated with spiking solutions. To eliminate these and other problems, some analysts spike blanks or matrices similar to the waste in the laboratory and ship them, along with sample containers to the field. This approach also has its limitation because the matrix and the handling of the spike are different from those of the actual sample. In all cases, the meaning of a low field-spik recovery is different to interpret, and these field spikes are not commonly used.

2. Standard Operating Procedures;

   a) Definition of objectives.
   b) Design of sampling plans.
   c) Preparation of containers and equipment (specific analytical methods).
   d) Maintenance, calibration and cleaning of field equipment.
   e) Sample preservation, packaging and shipping.
   f) Health and safety protocols.
   g) Chain of custody protocols.

3. Health and Safety;

a) Monitoring the health of field personnel.
b) Routine safety procedures.
c) Emergency procedures.

4. Chain-Of-Custody;

a) Sample labels-gummed or tags (minimum)
   1) Sample number.
   2) Name of collector.
   3) Date and time of collection.
   4) Place of collection.

b) Sample seal-if necessary (minimum requirements)
   1) Sample number.
   2) Name of collector.
   3) Date and time of sampling.
   4) Place of collection.

c) Recorded in logbook (bound)-safely stored (minimum requirement);
   1) Location of sampling point.
   2) Name and address of field contact.
   3) Producer of waste and addresses (if different from location).
   4) Type of process producing waste (if known).
   5) Type of waste (eg. sludge, wastewater).
   6) Suspected waste composition, including concentrations
   7) Number and volumes if sample taken.
   8) Purpose of sampling (eg. surveillance, contract number).
   9) Description of sampling point and sampling methodology.
   10) Date and time of collection.
   11) Collector's sample identification number.
   12) Sample distribution and how transported (eg. name of laboratory, UPS, Federal Express).
   13) References such as maps or photographs of sampling site.
   14) Field observation.
   15) Any field measurements made (eg. pH, flammability, explosivity).
   16) Signatures of personnel responsible for observations.

d) Chain-of-custody record;
   1) Sample number.
   2) Signature of collector.
   3) Date and time of collection.
   4) Waste type.
   5) Signature of persons involved in the chain of possession.
   6) Inclusive dates of possession.

LABORATORY QUALITY ASSURANCE

I. FACILITIES AND PERSONNEL:

A. Ventilation: Central-air, free of dust, drafts, and extreme temperature changes.

B. Space Utilization:
   1. Design and operate to minimize traffic.
   2. Provide a separate area for preparing and sterilizing media, glassware and equipment.
   3. Use a special work area such as a vented laminar-flow hood for dispensing and preparing sterile medi transferring microbial cultures or working with pathogenic materials. (In smaller laboratories it may be necessary, although undesirable to carry out these activities in the same room.)

C. Laboratory Bench Area:
   1. Provide at least 2 meter linear bench spaces per analyst, and additional areas for support and preparation areas.

D. Walls And Floors: Washable surface.

E. Air Monitoring

F. Laboratory Cleanliness:
   1. Regularly clean laboratory rooms and wash benches, shelves, floors and windows.
   2. Wet mop floors and treat with disinfectant solution do not sweep or dry-mop.
   3. Wipe bench tops and treat with disinfectant before and after.

G. Personnel:
   1. Professional microbiologist or trained analyst available for guidance and assistance.
   2. Clearly defined work assignments.
   3. Train the analyst in basic laboratory procedures.
   4. Review procedures of sample collecting and handling media and glassware preparation, sterilization, routine testing procedures, counting, data handling, and quality control techniques to identify and eliminate problems

II. LABORATORY EQUIPMENT AND INSTRUMENTATION:

Verify that each item of equipment meets the user's needs fo accuracy and precision. Perform equipment maintenance on a regula basis as recommended by the manufacturer, or obtain preventive mai tenance contract on autoclaves and balances whenever economically feasible. Directly record all quality control checks in a PERMANENT LOG BOOK.

A. Thermometer/Temperature Recording Instruments:

1. Check accuracy of thermometers or recording instruments semi-annually against a certified National Bureau Of Standards (NBS) thermometer, or one trace able to NBS and conforming to NBS specifications.
2. a) For general purposes use thermometers graduated in increments of 0.5° or less.
   b) For a 44.5°C water bath, use a submersible thermometer graduated to at least 0.2°C.
   c) Record temperature check data in a quality control log book.
   d) Mark NBS calibration corrections on each thermom eter used with an incubator, refrigerator or freeze
   e) If possible, put a temperature recorder on incubators and water baths.

B. Balances:

1. Wipe balance before and after each use with a soft brush made of such materials as camel hair.
2. Clean balance pans after each use and wipe spills up immediately with a damp towel.
3. Inspect weights with each use, and discard if corroded.
4. Check weights monthly against certified weight.
5. a) For weighing 2g or less, use an analytical balan with sensitivity less than 1mg at a 10g load.
   b) For larger quantities, use a pan balance with sensitivity of 0.1g at a 150g load.

C. pH Meter:

Standardize pH meter with at least two standard buffers (pH 4.0, 7.0, or 10.0) and compensate for temperature before each series of tests. Date buffer solutions when open and check monthly against another pH meter.

D. Water Deionization Unit:

1. Proper mixed-bed deionization resin columns produce a good grade of pure water.
2. Monitor deionized water continuously, or daily with conductivity meter and analyze for trace metals annually.

E. Water Stills

F. Reverse Osmosis Units

G. Media Dispensing Apparatus

H. Hot-air Oven:

1. Test performance with spore strips or spore suspension quarterly.
2. Monitor temperature with a thermometer accurate in 160° to 180°C range, and record results.
3. Use heat-indicating tape to identify supplies and materials that have been exposed to sterilization temperatures.

I. Autoclave:

1. Record temperature, pressure and time for each run.

2. Check operating temperature weekly with a minimum/ maximum thermometer.
3. Test with spore strip or suspensions monthly.
4. Use heat sensitive tape to identify supplies and materials that have been sterilized.

J. Refrigerator:
1. Check and record temperature daily and clean unit monthly.
2. Identify and date materials stored.

K. Freezer

L. Membrane Filter Equipment:
1. Before use, assemble filtration units and check for leaks.
2. Coat units with silicone to improve drainage.
3. Clean filtration assemblies thoroughly after use, wrap in nontoxic paper or foil, or place in non-corrosive container and autoclave.

M. Ultraviolet Sterilization Lamps:
1. Disconnect unit monthly and clean lamps by wiping a soft cloth moistened with ethanol.
2. Test lamps quarterly with UV light meter and replac if they emit less than 70% of initial output, or if agar spread plates containing 200 to 250 microorgan isms, exposed to the light for 2 min do not show a count reduction of 99%

N. Safety Cabinet (Hood)

O. Water Bath

P. Incubator (Air or Water Jacketed):
1. Check and record temperature twice daily (morning and afternoon).
2. If a glass thermometer is used, submerge bulb and stem in water or glycerine to the stem mark.

Q. Microscopes:
Use lens paper to clean optics and stage after each use Cover microscope when not in use.

III. LABORATORY SUPPLIES:

A. Glassware:
Each use, examine glassware and discard items with chipped edges or etched inner surfaces. Inspect glassware after washing, it water beads excessively on clean surfaces rewash. Make the following tests for clean glassware:
1. pH check - to test clean glassware for an alkaline or acid residue add a few drops of 0.04% bromothymo blue (BTB) or other pH indicator and observe the color reaction. Bromothymol blue may be yellow (acid) to blue-green (neutral) to blue (alkaline) i the pH range of 6.5 to 7.3. - To prepare 0.04% Bromothymol blue indicator solution add 16mL 0.01N NaOH to 0.10g BTB and dilute to 250mL with distille water.

2. Test for inhibitory residues on glassware - Do this test annually and before a new supply of detergent.

a) Procedure:

1) Wash six petri dishes according to usual laboratory practice and designate as Group A.
2) Wash six petri dishes as above, rinse 12 time with successive portions of distilled water and designate as Group B.
3) Rinse six petri dishes with detergent wash water (use in concentration), dry without further rinsing and designate as Group C.
4) Sterilize dishes in Groups A, B, and C by usual procedure. To test presterilized plasticware, set-up Group D, consisting of six sterile petri-dishes and proceed.
5) Add not more than 1mL of sample yielding 50 t 150 colonies and proceed according to the pro cedure described for the heterotrophic plate count. If there is difficulty in obtaining a suitable sample, inoculate three plates of each group with 0.1mL and the other three plates of each group with 1mL.

b) Interpretation Of Results:

1) Difference in average number of colonies of less than 15% on Groups A, B, C and D indicates that the detergent has no toxicity or inhibitory characteristics, or that the presterilized dishes are acceptable.
2) Differences in colony count of 15% or more be tween Group A and B or D and B demonstrates inhibitory residue.
3) Disagreement in averages of less than 15% between Groups A and B and greater than 15% between Groups A and C indicates that the clean ing detergent has inhibitory properties that are eliminated during routine washing.

B. Utensils And Containers For Media Preparation:

1. Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other noncorrosive an noncontaminating material.

C. Pure Water Quality:

The quality of water obtainable from a pure water system differs with the system used, and its maintenance. Acceptable limits of water quality are given in Table 1. If limits are not met, investigate and correct. Although pH measurement or purified water is characterized by drift, extreme readings are indicative of chemical contamination.

ANALYTICAL LABORATORY SERVICES
A Division of M C Chemical
P.O. Box 1926 · Rockford, Il 61110
815/964-7570

July 28,1992

Customer Name
Address
Rockford, Il 61111

Laboratory Sample # 920728001
Customer Description
Date Received 7-28-92

VOLATILES	ADL	Results
	mg/L	mg/L
Acetone	0.100	BDL
Acetonitrile	0.100	BDL
Acrylonitrile	0.010	BDL
Acrolein	0.005	BDL
Allyl Alcohol	0.005	BDL
Allyl Chloride	0.010	BDL
Benzene	0.002	BDL
Benzyl Chloride	0.100	BDL
Bromoacetone	0.010	BDL
Bromodichloromethane	0.001	BDL
Bromoform	0.005	BDL
Bromomethane	0.010	BDL
2-Butanone	0.010	BDL
Carbon Disulfide	0.100	BDL
Carbon Tetrachloride	0.005	BDL
Chlorobenzene	0.005	BDL
Chlorodibromomethane	0.010	BDL
Chloroethane	0.010	BDL
2-Chloroethanol	0.010	BDL
2-Chloroethyl Vinyl Ether	0.010	BDL
Chloroform	0.0005	BDL
Chloromethane	0.010	BDL
Chloroprene	0.005	BDL
3-Chloropropionitrile	0.010	BDL
1,2-Dibromo-3-Chloropropane	0.100	BDL
Dibromomethane	0.005	BDL
1,4 Dichloro-2-Butene	0.100	BDL

BDL-Below Detection Limit

ANALYTICAL LABORTORY SERVICES

US4657

A Division of M C Chemical
P.O. Box 1926 - Rockford, Il 61110
815/964-7570

Customer Name
Laboratory Sample #920728001

Customer Description:
Date Received: 7-28-92

VOLATILES	ADL mg/L	Results mg/L
Dichlorodifluoromethane	0.005	BDL
1,1 Dichloroethane	0.0007	BDL
1,2-Dichloroethane	0.0003	BDL
1,1-Dichloroethene	0.0013	BDL
Trans-1,2-Dichloroethene	0.010	BDL
1,2 Dichloropropane	0.010	BDL
1,3 Dichloro-2-propanol	0.010	BDL
Cis 1,3-Dichloropropene	0.010	BDL
Trans-1,3-Dichloropropene	0.005	BDL
1,2,3,4-Diepoxybutane	0.010	BDL
1,4 Dioxane	0.010	BDL
Epichlorohydrin	0.010	BDL
Ethanol	0.005	BDL
Ethyl Benzene	0.005	BDL
Ethylene oxide	0.010	BDL
1,2 Dibromoethane	0.005	BDL
Cis 1,2-Dichloroethene	0.005	BDL
Ethyl Methacrylate	0.005	BDL
2-Hexanone	0.050	BDL
2-Hydroxypropanenitrile	0.010	BDL
Iodomethane	0.005	BDL
Isobutyl Alcohol	0.100	BDL
Isopropylbenzene	0.010	BDL
Malononitrile	0.010	BDL
Methacrylonitrile	0.100	BDL
Methylene Chloride	0.005	BDL
Methyl Iodide	0.005	BDL
Methyl Methacrylate	0.050	BDL
4-Methyl-2 Pentanone	0.050	BDL
Pentachloroethane	0.010	BDL

BDL-Below Detection Limits

ANALYTICAL LABORATORY SERVICES

US4658

A Division of M C Chemical
P.O. Box 1926 - Rockford, Il 61110
815/964-7570

Customer Name
Laboratory Sample #9207280001

Customer Description:
Date Received: 7-28-92

VOLATILES	ADL	Results
	mg/L	mg/L
2-Picoline	0.010	BDL
Propargyl alcohol	0.010	BDL
b-Propiolactone	0.010	BDL
n-Proylamine	0.010	BDL
Propionitrile	0.100	BDL
Pyridine	0.010	BDL
Styrene	0.005	BDL
1,1,1,2-Tetrachloroethane	0.005	BDL
1,1,2,2-Tetrachloroethene	0.005	BDL
Tetrachloroethene	0.0003	2.354
Toluene	0.002	BDL
1,1,1-Trichloroethane	0.0003	BDL
1,1,2-Trichloroethane	0.0003	BDL
Trichloroethene	0.0012	BDL
Trichlorofluoromethane	0.005	BDL
1,2,3-Trichloropropane	0.005	BDL
Vinyl Acetate	0.050	BDL
Vinyl Chloride	0.0010	BDL
Xylene	0.0005	7.267

BDL-Below Detection Limits
EPA method 8240

Thank you

Pamela M. Fieser
Analytical Chemist

US4659